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Wood Veneer Reinforced with Bacterial Cellulose: Tensile Strength and Dynamic Mechanical Analysis

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***Abstract*— Cellulose from plants is a natural polymer that is very abundant, cheap and easy to process. In addition, there is bacterial cellulose produced from bacterial fermentation of acetic acid with limited production but has high purity, crystallinity, and tensile strength. In this research, the process of wood-veneer delignification was carried out to self-assembly bacterial cellulose into wood cavities on bacterial culture media. Wood veneer reinforced bacteria cellulose was given heat pressure to increase the density and it was expected to form hydrogen bonds between their cellulose molecular chains. This study observed the duration of fermentation in bacterial media culture on the tensile strength of hybrid veneers, microscopic observations, the effect of water on set-recovery, and the characteristics of solid veneers on cyclic loading and temperature using dynamic mechanical analysis (DMA) testing. The microscopic observations prove that Acetobacter xylinum can penetrate the veneer and assemble bacterial cellulose in the cavity. A higher tensile strength ratio of 81.38% was observed in densified veneers with a five-day fermentation period with a modulus of elasticity 156.63% higher than natural veneers. The minimum set-recovery after boiling the hybrid veneer was 29.32%. DMA showed that by reinforcing wood veneer with bacterial cellulose and compacting, it increased cyclic energy storage ability, reduced energy loss, and increased stability under increasing temperatures. Strengthening wood with bacterial cellulose in this method opens new potential for developing more environmentally friendly forest products in the future.**

***Keywords*— Delignification; cellulose; bacterial; densified; veneer; hydrogen bonds; tensile strength.**

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I. INTRODUCTION

Platinum Teak Wood (*Tectona grandis* L.f.) is a fast-growing tree that the Indonesia Institute of Sciences has developed. Berlian Teak Wood developed it through the tissue culture method and radiation treatment. Based on macroscopic and microscopic, the anatomy of Platinum Teak wood has similar characteristics with others Teak wood conventional or Teak wood from the community forest and have semi-ring porous which the size changes gradually from earlywood to latewood [1]. This wood has good prospects for use. The physical properties of Platinum teak are better than teak wood from a community forest. Even though the dimension stability of Platinum teak is low, the mechanical property is similar to the conventional teak at a class age of 20-30 years [2].

The densification process without chemicals is known as the improvement of physical properties, which can improve wood's density and mechanical properties. The wood-densification method needs a pressure force and hot temperature. Wood density can be improved by compression, but it is not easy to reach a fixation phase in the wood. In the wood industry, several methods for deformation fixing of densified wood include high-temperature steam, resin impregnation, and high-frequency microwave heating [3]. The delignification process is the first step and a new way of the wood-densification method. This process can improve high mechanical strength with temperature treatment at 100ºC [4]. Hydrogen bonding occurring in the delignification of nanofibers can be induced by water molecules during the drying and compressing. Thus it affects the physical and morphological properties [5]. The mechanical properties of the Pine densified veneer increased along with giving heat pressure at 150ºC, but the mechanical properties decreased if the temperature was increased [6].

Acetobacter xylinum has the coating ability of natural fibers with bacteria cellulose as additional reinforcement to increase mechanical strength [7]. From some studies, the interaction between bacteria cellulose and plant cellulose has the potential to make strong hydrogen bonding between the cellulose chains [8]. The utilization of bacteria cellulose on natural fibers can improve surface bonding between natural fibers and polymer adhesive [9]. The film product of bacteria cellulose can still be degraded for 5-6 weeks in the soil, making it environmentally friendly [10]. The previous study did not explain the ability of bacteria to penetrate more complex and thicker fibers, such as wood, to form pure cellulose. However, a study shows that bacteria cellulose can fill between the softwood fibers on thin paper sheets [11].

Due to the hygroscopic nature of wood, when densified wood is exposed to humidity or water for a certain period, wood can return to its original shape. It remembers its earlier shape, and it is always possible to return to its original shape. Recovery to the original shape can occur as long as the compaction process of a crystalline area of microfibers has elastic deformation. The energy release of elastic strain in cellulose macromolecules can be considered a cause of set-recovery [12]. On the other hand, spring-back is a natural reaction of wood to release the stresses generated during an operation. The spring-back effect occurred quickly after the stress that caused the deformation was removed, then it resulted immediately set-recovery in the sample [13].

During utilization and application, the product of wood technology, such as densified wood, will be influenced by the dynamic load. As a result, it is critical to understand and research the performance of densified wood under periodic force. A dynamic mechanical analyzer (DMA) is a technique to give information about thermo-mechanic properties of the material on the dynamic load is a dynamic mechanical analyzer (DMA). DMA was used to describe structure and attenuation as frequency function, time, pressure, or combination of the parameters. DMA also depends on the physical composition or the structure of the phase, such as interface, morphology, and constituent properties [14].

In this research, the delignification veneer was assembled with bacteria cellulose with assembly in the culture medium. Then the hybrid veneer was densified with heat treatment. This method was used to improve the physical and mechanical properties of the veneer. The observation used a microscope to determine and demonstrate the ability of bacteria to penetrate veneer pores. A tensile test was used to know the mechanical strength of the compaction veneer. Loss of weight and set recovery were used to determine the dimensional stability of densified veneer in the face of environmental influences. On the other hand, the DMA technique was used to determine the effect of dynamic load against storage modulus (E') and damping factor (tan δ) on the veneer sample, which is affected by increasing temperature.

II. MATERIALS AND METHOD

A. Materials

This research uses Platinum teak (*Tectona grandis* L.f), 9 years old, which was obtained from Cibinong Science Center, Bogor. This wood was cut into a log and then taken from the center of a 120 cm long and 38 cm diameter wood. The log is then cut into 2.3 mm thick veneer sheets using a rotary machine. The veneer sheet was then cut into a 10 x 10 cm sample. All samples were free from knots and visible defects.

The other materials were a starter to produce bacteria cellulose. The main process for producing the bacteria cellulose was the fermentation of coconut water with the final product known as nata de coco. The starter or cell bacteria used in this study was *Acetobacter xylinum*. *Acetobacter xylinum* is a gram-negative bacteria that can polymerize glucose into cellulose strands [15]. *Acetobacter xylinum* has 0.5-1 μm width and 2-10 μm length, whereas the bacteria produce cellulose fiber with 30 – 80 nm width [16]. The cells were bought from Biotechno Store Collection in liquid form.

B. Methods

1) Delignification: The Wood delignification process is to dispose of a little lignin and hemicellulose in wood. Cellulose is a strong natural polymer with difficulty in the degradation process even if it uses the strongest NaOH solution. In previous research, the delignification process of Platinum teak has produced compacted wood with good strength if the veneer boiled for 90 minutes [17]. Therefore, the veneer sample in this study was boiled in 1 M of NaOH solution using a beaker glass at 90ºC for 90 minutes. Boiling for 90 minutes was also ideal for converting teak veneer to compacted wood with good mechanical properties [17]. However, some samples were boiled at 30, 60, 90, 120, and 150 minutes to analyze their chemical wood content. This testing was to know the percentage of degradation of lignin and hemicellulose. The Chesson-Datta method was used to determine the chemical wood content, such as cellulose, hemicellulose, and lignin [18]. After boiling, veneer samples were rinsed using water. The residual of the NaOH solution and other substances on the wood was disposed of by soaking in clean water for 24 hours. Then, veneer samples were rinsed again with clean water.

2) Self-assembly of bacteria cellulose: The material composition consists of 5% white sugar, 1% acetic acid, and 0.1% urea for 1 L of coconut water. First, filter coconut water with clean fabric and boil it on the stove. Second, reduce stove fire and put acetic acid gradually until pH 4.5. Third, stirred urea and white sugar, then stirred the solution and boiled on low heat for 15 minutes. The next process was the assembly of bacteria cellulose into delignification of wood veneer. In culture media, bacteria cellulose was assembled into veneer fiber. First, the veneer sheet was placed in a plastic box, and then hot culture media was poured into the plastic box until the veneer was completely saturated. Covered the box with sterile paper immediately to prevent contamination from the air. Wait until the temperature reaches 30°C before adding 10 mL of bacteria starter for 1 L of culture solution. Starter bacteria should be added to culture media quickly, with the paper cover slightly open. Then culture media was conditioned at room temperature 28-30ºC. The duration of fermentation was 5, 6, 7, and 8 days. After that, veneer and pellicle bacteria cellulose could be harvested to become a hybrid veneer.

3) Densified wood: Wood veneer, harvested from culture media, was rinsed with clean water, then this hybrid veneer was drained and conditioned until the water content was 30%. The water content was measured by a Digital Pin Moisture Meter. Furthermore, the veneer was densified using a compression machine at temperature 115ºC and pressure of

10 MPa by gradually increasing pressure of 2.5 MPa every 15 minutes for 1 hour. The veneer was then drained into the oven, and a steel plate loaded the sample's condition. The temperature of the oven was 70^{0}C for 2 hours or until the water content of the veneer was 0-1%. The veneer was then compressed again for 1 hour at 135^{0}C with a pressure load of 10 MPa. Afterward, the veneer sample was taken and conditioned at the desiccator.

4) Microscopic observation: microscopic observation was needed to look at the surface in the cross-section of the hybrid veneer and to confirm the assembly can be done by an in-situ process. After being harvested from culture media, the 10 x 10 cm veneer sample dimension was cut into 3 pieces. These pieces were used to observe the surface of the veneer using a microscope (KEYENCE, VHX-6000). The observed samples were taken from every veneer sheet. There were two sections at the edge and one section in the middle, as shown in Figure 1. For the presentable surface of cutting, the samples were cut using Microtome.

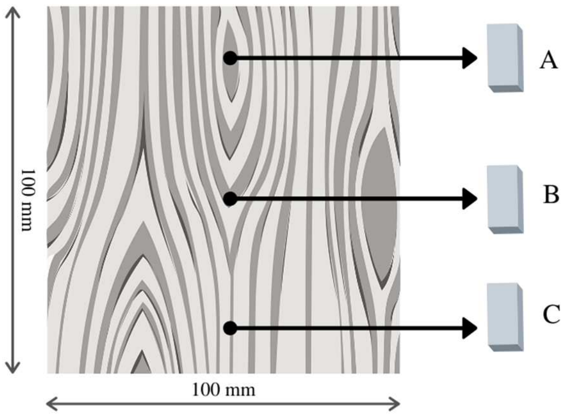

Fig. 1 Sampling position of veneer sheet.

5) Tensile strength: A Tensile strength test was conducted to know the mechanical strength of densified veneer, both before and after treatment assembly bacteria cellulose in situ. In comparison, this research tests the tensile strength of the natural teak veneer (NV), delignification-densified veneer (DDV), delignification-densified hybrid veneer (BDV) with the variations of period fermentation were 5, 6, 7, and 8 days. All samples of tensile strength were 10 mm in length and 10 mm in wide and the number of test replicates was six samples [19]. Tensile strength testing was performed at room temperature using a UTM machine (SHIMADZU, AGS-X-10 kN) at a 0.6 mm/minute speed.

6) Set-recovery: Wood was affected by water and humidity in its surroundings because that densified wood product tends to spring back if the fixation process has not been finished [20]. The number of set-recovery (SR) in densified wood products with a heat press is computed using Equation 1 [20]:

$$SR = [(Tr - Tc)/(To - Tc)]x100\% \qquad (1)$$

where

SR : set-recovery (%);
To : the initial thickness of dry wood (mm);
Tc : the thickness of wood after compressed and dried at temperature 103±2^{0}C for 24 hours (mm);
Tr : the thickness of wood after boiled for 30 minutes then dried at temperature 103±2^{0}C for 24 hours (mm).

7) Analyze of dynamic mechanical: Analyzed dynamic mechanical used Perkin Elmer DMA 8000 with single cantilever bending mode. The dimension of the veneer was 10 mm in width and 40 mm in length with the range between 2 points of clamping was 6.5 mm in the middle of the span as shown in Figure 2. The sample was heated at 20^{0}C to 250^{0}C and the rate of temperature rise was 5^{0}C/minutes. Moreover, the strain load was 0.01 mm with a fixed frequency of 1 Hz. From DMA, we got information about storage modulus, loss modulus, and damping factor (tan δ) which depended on the temperature. DMA gave information about glass transition temperature (Tg).

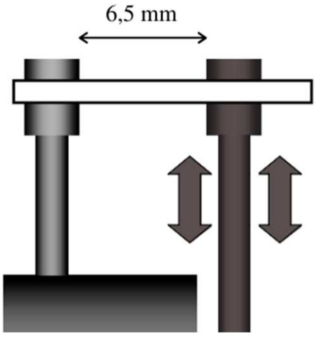

Fig. 2 DMA single-cantilever bending mode

III. RESULTS AND DISCUSSION

A. Delignification and Chemical Component of Veneer

In this research, we used 9 years of Platinum teak with a position in the middle of a wood. This section has 13.4% of hemicellulose, 31.5% cellulose, and 22.7% lignin. The NaOH delignification process showed that hemicellulose and lignin degraded during the boiling process. On the other hand, cellulose content still stayed on the veneer as shown in Table 1. The result was similar to Song's research, where the biggest strength of compacted wood was obtained by removal of lignin up to 11.3% - 15.1% [4].

TABLE I
CHEMICAL CONTENT OF WOOD VENEER BY BOILING TIME OF SODIUM HYDROXIDE.

Time of delignification	**Hemicellulose (%)**	**Cellulose (%)**	**Lignin (%)**
Natural	13.40	31.50	22.70
30 minutes	12.60	32.70	20.60
60 minutes	10.30	34.80	18.50
90 minutes	8.80	35.40	15.60
120 minutes	8.10	36.20	14.70
150 minutes	7.40	37.20	13.30

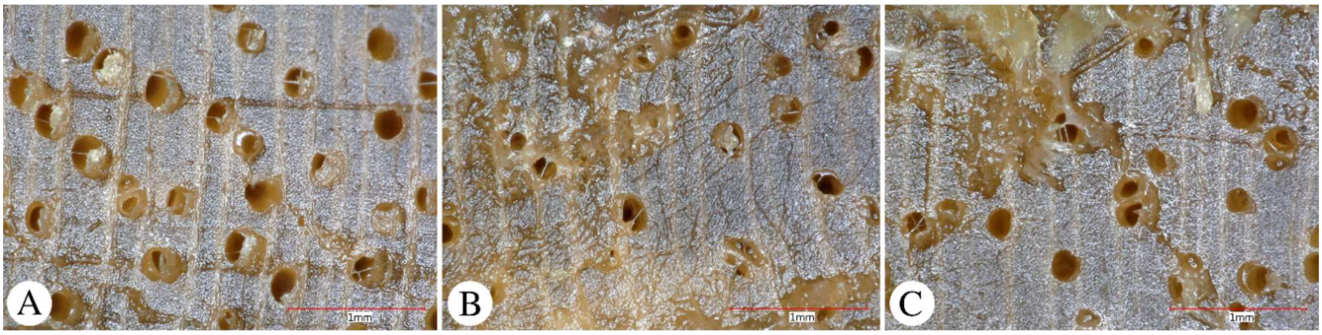

Fig. 3 Cross-section of veneer after harvesting from growth media (A) edge (B) middle (C) other edges

TABLE II
PHYSICAL AND MECHANICAL PROPERTIES OF VENEERS

Sample	Density	Tensile Strength	Modulus Elasticity	Set-Recovery	Weight Loss
	(g/cm^3)	(MPa)	(MPa)	(%)	(%)
NV	0.62	78.92 ± 14.31	5741.24 ± 575.88	-	-
DDV	1.00	110.89 ± 20.50	11976.59 ± 885.84	48.95	17.28
BDV-5	1.18	143.15 ± 49.55	14733.97 ± 1666.49	29.32	16.01
BDV-6	1.18	138.78 ± 39.11	14203.52 ± 709.96	35.34	15.66
BDV-7	1.19	131.39 ± 27.53	14660.00 ± 1019.07	36.44	15.53
BDV-8	1.23	124.07 ± 49.29	12477.96 ± 720.20	37.40	14.46

B. Bacteria Cellulose in the veneer cavity

Figure 3 shows that bacteria cells could penetrate the wood on all sides until the 2.3 mm thickness after the delignification process. From the ImageJ software, the diameter of wood pores was 0.12 to 0.35 mm. Bacterial penetration occurred because *Acetobacter xylinum* had a rod-shaped diameter that was smaller than wood pores. *Acetobacter xylinum* had 0.5-1 µm in width and 2-10 µm in length, whereas the bacteria could produce cellulose fiber which was 30 – 80 nm in width. Moreover, natural fiber properties, like hydrophilic with a rough surface, were ideal for growing *Acetobacter xylinum* [21].

Nevertheless, it showed in a microscope that the penetration had not covered the pores in the wood. However, the fulfillment bacteria cellulose occurred in all sections of wood where it was soaked in culture media. There was a strong interaction between bacteria cellulose and wood fiber as the cellulose has a strong affinity against itself and another material that contains a hydroxyl group [22]. Bacterial cellulose fills and covers the pores of wood veneer, and this occurs thoroughly near the wood surface. The process was self-explanatory because *Acetobacter xylinum* is a typical aerobic metabolism bacterium that requires oxygen to grow [23].

C. Tensile Strength and Set-recovery

Heat compression on the delignification of the veneer was divided into two phases, i.e., wet and dry conditions. Heat compression in the wet condition could easily compact and produce a good, densified wood and it was not damaged. During the compression process, heat compression in the dry condition was expected to have a vital role in connecting nanocellulose fibers. All water content was not wasted when the veneer was compressed under heat in wet conditions for 1 hour. The remaining water content in the veneer was 1-4%. There was no spotted water in the veneer and not all water could be evaporated during heat compression. After drying in the oven for 2 hours at 70^{0}C, the rest of the water in the veneer could be evaporated. The veneer was in a temporary state at this point because, when immersed in water, it will return to 75 to 85% of its original size. In the second phase, the veneer was compressed in dry condition. We expect that mechanical properties will increase and approach fixation after heat compression treatment in this phase.

Table 2 shows that delignification-densified veneer (DDV) has a higher density and improves the tensile strength by around 40.5% than natural veneer (NV). The modulus elasticity of the densified veneer increased up to 108.6%. Delignification veneer caused lignin to be partially degraded. As a result, compact wood cell walls create densified wood that is denser than natural wood. The higher density of cellulose fibers resulted in hydrogen bond interaction on the cellulose molecular chain, which increased tensile strength [4].

Table 2 shows that tensile strength and elastic modulus increase on bacteria cellulose reinforced veneers. It shows that bacteria cellulose acts as additional reinforcement to increase the mechanical properties of compacted veneers. Bacteria cellulose and natural fiber cellulose have the hydroxyl chain on the cellulose molecule chain. These hydroxyl chains would interact strongly if they had been close. Then this occurrence has the potential to increase the formation of hydrogen chains. [24][25]. Tensile strength and elastic modulus of bacteria cellulose reinforced veneers with fermentation for 5 days (BDV-5) were highest. There was an increase in tensile strength up to 81.38% and elasticity modulus up to 156.63% on natural veneers. However, tensile strength and elasticity modulus decreased when the durations of fermentation were 6, 7, and 8 days. However, this decrease was not significant.

The length of fermentation time caused a decrease in the tensile strength of the bacteria cellulose reinforced veneer. Set-recovery data explained this. The density increased and an increase followed it in set-recovery. This is due to the increase in the number of pellicles during fermentation time, causing not all bacteria cellulose to interact and bond with wood cellulose. After densified, there was a free hydroxyl chain of bacteria cellulose, and this chain had an interest in water which is affected by the humidity of the surrounding air. Water in the pellicle will work as a plasticizer. This causes a decrease in the number of hydrogen bonds between molecules and then changes their mechanical properties [26]. Similarly, the hydroxyl groups on the molecular chains of wood cellulose which bind to water, cause a decrease in mechanical strength.

From Table 2, we can see that the longer assembly time of bacteria cellulose in media growth caused a decrease in the percentage of weight loss (WL) in veneer. This demonstrated the formation of a pellicle of bacteria cellulose in the pores of the veneer during fermentation. As a result, the weight loss in veneer after compression decreased because of the escalation of bacteria cellulose. Weight loss and set-recovery in densified wood had a correlation, which is the greater weight loss caused, the smaller set-recovery [27]. This correlation was appropriate for this study because weight loss would increase set-recovery. Weight loss in densified wood is affected by temperature during compression. Increasing the number of pellicle bacteria cellulose during fermentation time in veneer needed high temperature on compression. This is intended to decrease set-recovery.

D. Dynamic Mechanicals Analyze

Dynamic mechanical analysis (DMA) is a technique measurement material stiffness as a function of time, temperature, and frequency when the material encounters the controlled atmosphere. DMA applies deformation sinusoidal periodic on the sample with a small amplitude. This sample will respond to it by showing strain and attenuation such as storage modulus and tan δ. The storage modulus is known as modulus Young. It relates to the stiffness of the material and determines the thickness and stiffness of a sample. Storage modulus is considered as the material's ability to store the energy.

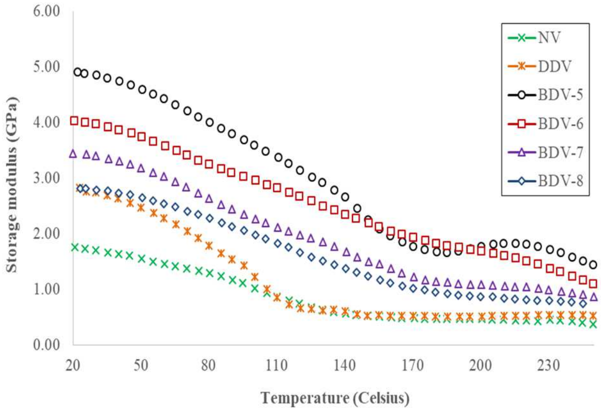

Fig. 4 Effect of temperature to storage modulus

The effect of temperature and storage modulus in Figure 4 shows the similarities and differences in response between natural veneer (NV), delignification-densified veneer (DDV), and delignification-densified hybrid veneer (BDV). The similarities are that the storage modulus decreases as the heating temperature rises due to increased chain mobility of the polymeric component of the wood cell wall. Meanwhile, the difference is in the initial storage modulus and others, in which the bacteria cellulose reinforced veneer with 5 days of fermentation (BDV-5) had the highest storage modulus. Subsequently, it was followed by a decrease in the initial storage modulus on BDV-6, BDV-7, and BDV-8. However, the storage modulus of BDV was higher than NV and DDV. The response level of storage modulus on every BDV had the same mechanical strength properties such as tensile strength on the previous veneer. Bacteria cellulose is more crystalline and has strong hydrogen bonds. The crystalline area acts as a physical cross-linker, like a filler particle that increases storage modulus.

Figure 4 shows that all BDV samples had the same response to the storage modulus, but the DDV sample had a different response. DDV samples had a higher storage modulus than NV samples. However, the response was like the storage modulus when the temperature reached over 115ºC. This was due to the glass transition at temperature 115ºC-150ºC [28]. A higher initial storage modulus in the DDV sample was supported by density and hydrogen chains between the wood fiber cellulose, but these chains were weak. When these chains enter the glass transition area, they are released without additional reinforcement. Therefore, the ability of storage energy would be back to the natural veneer. This showed that the existence of bacteria cellulose was important to improve and maintain the storage modulus in veneer during the increase in heating temperature.

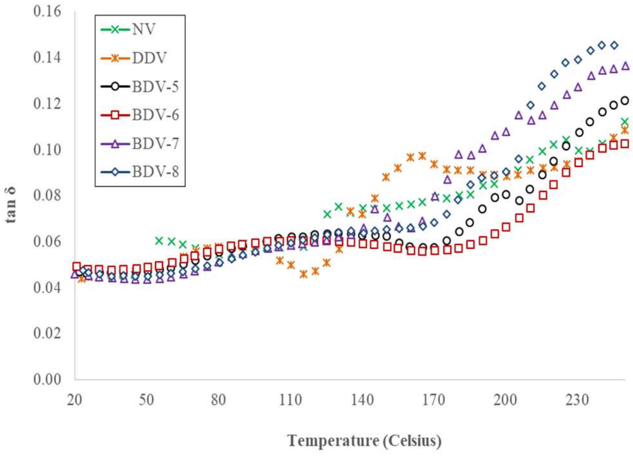

Fig. 5 Effect of temperature and tan δ

Damping factor (tan δ) is a ratio of loss modulus and storage modulus. Loss modulus is a material response that is considered to be a material's tendency to eliminate the energy applied to it. Figure 5 shows the effect of temperature and tan δ. It indicated the same damping behavior for all veneers. In general, the ability to absorb energy decreases as the heating temperature rises. NV and DDV had a lower and fluctuating damping ability, while BDV had a higher and stable damping ability until 180ºC. If it was seen in more detail, the damping factor of all BDV had the same value and a characteristic of under 115ºC. However, the damping response of every treatment started to change after it reached the peak of glass transition at 115ºC. All BDV samples were easily recognized as polymer behavior, with a glassy state occurring at room temperatures to 60°C and glass transition (Tg) occurring at temperatures of 60°C-150°C. While the rubbery state happened at 150°C-180°C and the veneer entered the viscous flow region at above 180°C. This temperature range had compatibility with the DMA test on the wood and the composite veneer [29][28]. Moreover, the DMA test on bacteria cellulose reported that temperatures of 60°C-110°C could be associated with glass transition temperature [30].

IV. CONCLUSION

Veneer reinforced bacteria cellulose requires an optimum delignification process from alkaline solution concentration, temperature, and duration of boiling time. In this study, the delignification process of veneer with 2.3 mm of thickness used 1 M of NaOH and 90°C in temperature. It required a minimum immersion time of 90 minutes and resulted in less than 15.6% lignin degradation. The delignification process resulted in the penetration of bacterial cells into the wood cavity. Then the penetration of bacteria cellulose caused the wood cavity to close by bacteria cellulose nano-fibers during immersion in the bacteria media culture. The maximum ability of bacterial cells to penetrate the delignification wood cavity until a certain thickness was unknown and was an interesting topic for future research.

Bacteria cellulose and wood fibers in the veneer that were densified with the heating press process, purposed as an additional reinforcement to increase the mechanical strength. This method rose the tensile strength of natural veneer up to 81.38% and the modulus of elasticity up to 156.63%. Five days of assembly bacteria cellulose on bacteria media culture showed the best performance, increasing mechanical strength and reducing set-recovery. The longest assembly decreased mechanical strength. On the other hand, it increased set recovery, although it was insignificant. From the DMA result, we conclude that reinforced bacteria cellulose (hybrid veneer) was the best storage cyclic energy, the highest reducing energy, and the most stable of the damping than natural veneer and densified ordinary veneer.

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CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

All authorships are main contributor. Ananto Nugroho: conceptualization, investigation, methodology, writing original draft. Triastuti: writing, review & editing. Sandi Sufiandi: review & editing. Anne Zulfia Syahrial: funding, review & editing.

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