Performance of Binderless Board Manufactured Using Camphor Tree Residue

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Residue of the branch wood of camphor tree (*Cinnamomum camphora*) was used to manufacture binder-free biodegradable biomass fiberboard (bio-board) by the wet method. To investigate the mechanical properties of the bio-board, bending rupture stress and tensile rupture stress tests were measured and the effect of heating temperature and applied pressure on the performance of the bio-board was evaluated. Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were used to analyze the influencing factors on the microstructure and bond quality of the bio-boards. The overall density of the bio-boards exceeded 0.8 g/cm^3 and the moisture content was below 10%, which conformed to the JIS A 5908 standard (2014) for the hardboard S20 type design specification. The bending rupture stress and tensile rupture stress continued to increase, and the dimensional stability continuously improved as the applied pressure and the heating temperature increased. However, when the heating temperature exceeded 170 °C, the increase in the rupture stress slowed down and the dimensional stability had been improved. Furthermore, increasing the heating temperature was more conducive for optimizing the bio-board's performance than increasing the applied pressure.

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INTRODUCTION

With the development of the world economy, wood has long been used as the first material for construction applications. However, the timber market is facing increasing demand between the construction and energy sectors. Usage of alternative materials such as natural fibers and biomass to replace wood in wood-based industries has attracted a lot of attention in recent years due to the depletion of forest resources and the consequent shortage of wood supplies.

At the same time, the use of healthier, safer, and more environmentally friendly materials is becoming a priority for the construction industry. In this context, the use of forestry waste as a raw material to replace wood seems to be a promising solution, especially due to the wide variety of lignocellulosic resources available worldwide (Mahieu *et al.* 2019; Lee *et al.* 2020). The global production of wood biomass wastes is 4.6 Gt per year, of which 20% is production loss (Tripathi *et al.* 2019).

Although these forest residues are used for animal husbandry, bio-manuring, soil cover, temporary shelter, and fuel for domestic and industrial purposes, a large proportion remains unused. Therefore, forestry residue and its bioproducts from agro-industries are a high research priority. In development of biodegradable composites such as fiberboards and particleboards, synthetic adhesive is used routinely for the preparation of building materials and for structural purposes (Nasir *et al.* 2019). Presently, most adhesives used in particleboards manufacture are formaldehyde-based, mainly urea-formaldehyde (UF) resins. Formaldehyde is a chemical compound classified as a human carcinogen and obtained from non-renewable resources. In the particleboards sector, formaldehyde is mainly released into the environment during resin production and the manufacturing of wood panels. After boards are produced, there is still a residual release of formaldehyde (unreacted and from UF hydrolysis), which makes formaldehyde-based resins a public health concern (Ferreira *et al.* 2019).

Ferrández-García *et al.* (2018) used UF as a binder to produce particleboard and found that particleboard could be made from almost all types of wood when the binder was added. However, toxic substances such as formaldehyde that are released during the use of particleboard are harmful to human health and the environment. At the same time, the binder cannot be released directly into nature after use, as it is non-degradable and is not in line with the main theme of sustainable human development. Therefore, the development of binderless particleboard has become an important focus.

Sahin *et al.* (2017) produced polymer composite particleboard using phenolic resin as the polymer binder. The effects of the forming pressure and the heating temperature on the properties of the particleboard were also investigated. Although both the forming pressure and the temperature can improve the particleboard properties, the phenolic resins also contain harmful substances such as formaldehyde. Therefore, the aim of this study was to develop the bio-board without binder to replace conventional particleboard. Unlike conventional particleboard, binderless fiberboard is manufactured by relying on the raw materials' internal bonding mechanisms rather than synthetic binders. Previous studies have found that as a cellulose-based structure is dried, it can contribute to hydrogen bonding and therefore enhances the mechanical properties of the board (Hubbe 2006; Halvarsson *et al.* 2009). Lignin is also present in a large proportion of tree cells and acts as a binder and reinforcer in cell wall formation (Kumar *et al.* 2009). Anglès *et al.* (2001) evaluated the effect of lignin composition on panel properties and demonstrated that lignin significantly improved the mechanical properties.

In this study, bio-board was made from camphor tree (*Cinnamomum camphora*) residue as a forest waste source by a wet method and hot-press molding. To improve the inter-fiber bonding ability and the mechanical properties of the bio-board, it is necessary to evaluate the performance of the camphor tree residue bio-board under the different production conditions of molding pressure and heating temperatures. The mechanical properties of the bio-boards were evaluated using tests such as the bending strength and the tensile strength. In addition, the water resistance of the bio-boards was evaluated by the surface wettability test. The magnitude of its correlation with the dimensional stability test was discussed. The thermal stability, chemical changes, and microstructural changes of the bio-board under different conditions were investigated using thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

In this study, the residue of the camphor tree, the most common and important tree species in the subtropical broadleaf evergreen forest, was selected as the test material. The camphor tree can grow over a range of 10° to 30° (northern latitude) and thus belongs to the "sun-plant" species. The camphor tree has a fast growth rate and it is characterized by its fine and beautiful form, and its resistance to disease, insects, pollution, and acid rain (Wei 2003). It is one of the principal tree species for afforestation and a valuable timber species (Li *et al.* 2007). Camphor trees are usually planted in gardens or campuses as ornamental plants. The regular composition of the camphor tree includes 43.0% cellulose, 25.1% hemicellulose, 22.7% lignin, and 0.12% ash. In general, from the higher percentage of chemical composition than other species of trees, it can be inferred that camphor has a more promising future as a renewable resource in composite materials and other industrial applications (Fei *et al.* 2005).

Thermogravimetric Analysis

Since this paper investigated the effects of the heating temperature and the applied pressure on the properties of the bio-board, TGA was performed to measure the mass of the experimental material as a function of the heating temperature before setting the production conditions. The TGA of the raw materials was performed using a Mettler Toledo TGA 2 device (Columbus, OH, USA). Twenty mg of dried forest residue camphor tree fiber was placed in the TGA 2 alumina crucible and heated under a nitrogen atmosphere between 25 °C and 700 °C at a heating rate of 10 °C/min.

Manufacturing of Bio-board Samples

This study employed a wet process for making bio-boards, with production methods including cutting, soaking, refining, and forming. All four parts were prepared by physical means and therefore did not destroy the constituent components of the camphor branch. Furthermore, no chemical additives such as hydrophobic agents and binders were added during the production process.

First, an electrical disintegrator (SU16; Cowa Cutter Co., Shizuoka, Japan) was used to slice the branch of the camphor tree into tiny chips shorter than 10 mm that met the size requirements for use in the refining machine in subsequent experiments. After that, the tiny chips were soaked in water at a temperature of 20 ± 1 °C for three weeks, which caused the fibers in the tiny chips to soften and swell. Then the softened tiny chips of camphor were placed simultaneously with water in a refining machine (Model A Beat finer; Satomi Corp., Shizuoka, Japan), using the water as a circulation medium for refining. During the refining process the refining machine was run at a concentration of 4% to 6%, a speed of 1750 rpm, a water pressure of 0.4 MPa and a flow rate of 10 L/min. The process was carried out by physically grinding the camphor tiny chips to be dissociated into individual fibers, while cutting the length of the individual fibers to less than 2 mm. By adding water to obtain an aqueous fiber solution with a concentration of 0.05 g/mL, the resulting fiber solution met the requirements of the wet fiberboard process. Using a pH meter, the pH of the aqueous fiber solution was measured to be 7.81, which is weakly alkaline.

Finally, 400 mL of fiber solution was filled into a mould of 100 mm in length and width, and a hot press (IMC-180C; Imoto Machinery Co., Kyoto, Japan) with a manually controlled hydraulic press system was used to heat the fiber solution while applying

pressure to produce the bio-board. During the forming treatment, pressure and temperature were applied simultaneously. The applied pressure was responsible for separating the water molecules bound to the fiber molecules, bringing the fiber molecules in intimate proximity with neighbouring fiber molecules linked by hydrogen and hydroxide bonds to form new chemical bonds, and then evaporating the water by adding heat to remove the water.

The experimental conditions for the manufacturing bio-boards are shown in Table 1, with a controlled mass of 20 g per bio-board. Depending on the production conditions, the thickness of the bio-board may vary, being around 2 mm. This study followed the industrial production process for the production of bio-boards, with each step being completed independently, and the performance advantages of the bio-boards under different conditions were investigated by varying the two test conditions.

Table 1. Experimental Conditions of Manufacturing the Bio-board

Experimental Condition	A	B
Applied pressure (MPa)	2.0, 3.5, 5.0, 6.5, 8.0	8.0
Heating temperature (°C)	110	110, 140, 170, 200, 230
Hot-pressure time (min)	120	30

Scanning Electron Microscopy Micro Analysis

The alignment of the bio-board internal and surface fibers bonding was observed *via* SEM. First, the bio-board was cut into 0.5 cm × 0.5 cm samples using an ultrasonic cutter (ZO-40B; Honda Plus, Shinshiro, Japan), in which a smooth surface cross-section could be created. The micro-structure of the biological plate was then observed using a Sigma 300 instrument (Carl Zeiss AG, Jena, Germany) in vacuum mode with a Gemini lens that could detect secondary electrons (SEs) and backscattered electrons (BSEs).

Spectroscopic Analysis

To classify the variety of functional groups that occurred in the samples from the produced bio-boards, FTIR spectroscopy analysis was conducted. The bio-board was cut into 1 cm × 1 cm samples. Then, the experimental samples were analyzed by a Nicolet iS50 FTIR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) with a resolution of 4 cm^{-1} between 4000 and 400 cm^{-1}.

Analysis of Physical Characteristics

The density of each bio-board sample was calculated by dividing the mass by the volume of the bio-board. Furthermore, the moisture content of the produced bio-boards was measured. The bio-boards were dried in a constant temperature dryer at 110 °C for 24 h to remove the moisture. The moisture content was calculated from the change in the weight of the bio-board samples.

Analysis of the Mechanical Properties

The physical properties of the bio-boards were evaluated for application in a broader range of fields. In compliance with the JIS A5908 standard (2014), the bending rupture stress and the tensile rupture stress values of the bio-boards were measured. Eight static bending rupture tests and six static tensile rupture tests were carried out on bio-boards under the same conditions. All tests were conducted using a universal testing machine (SVZ-200NB-200R2; IMADA Co., Toyohashi, Japan).

Water Affinity Analysis

The contact angle is a parameter that defines the wettability of materials and determines the waterproof performance of the material surface (Monroy *et al.* 2019). A pipettor was used to draw and dispense 5 μL of sterile distilled water on the surface of the bio-board. Images of the water droplets were taken by a high-definition digital microscope (NDL-40Z; Hirox Corp., Tokyo, Japan). Image processing was carried out using ImageJ (open-source software) to calculate the contact angle of the bio-board.

Analysis of the Dimensional Stability Properties

To explore the dimensional stability of the bio-boards, the water absorption (WA), longitudinal elongation (LE), and thickness swelling (TS) properties of the bio-boards were measured by WA tests. Specimens with dimensions of 50 mm × 50 mm were cut from the bio-board according to the JIS A5908 standard (2014). The specimens were immersed in water 2 cm below the water surface and placed in an incubator at a temperature of 20 ± 1 °C and relative humidity (RH) of 65 ± 1% for 24 h. The mass, longitudinal length, and thickness of the bio-board specimens were measured before and after the water absorption test and used to calculate the WA, LE, and TE values.

Statistical Analysis

The average values of duplicates were recorded for all the results. One-way analysis of variance (ANOVA) was used to test the experimental results, and $p < 0.05$ was rendered statistically insignificant.

RESULTS AND DISCUSSION

Thermogravimetric Analysis of the Materials

The TGA results of the dried residue from the camphor tree powder are shown in Fig. 1. The weight loss curve (TG) and the derivative thermogravimetric curve (DTG) are presented.

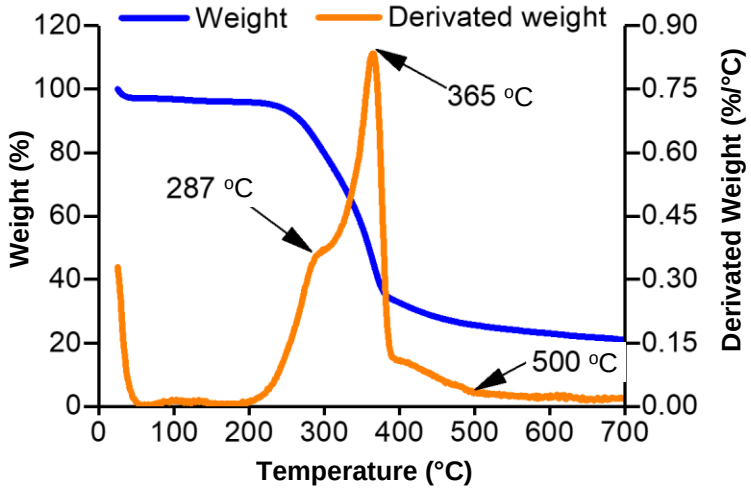

Fig. 1. TG and DTG curves of the materials

When the temperature was below between 25 and 50 °C, the thermogravimetric curve initially decreased. This weight loss may have been caused by the evaporation of water from the camphor tree residue during heating in a nitrogen atmosphere. There was little change in the weights between 50 and 200 °C. From 200 to 390 °C, the weight loss decreased from 95.58% to 33.79%, and the derivate weight loss rate achieved the maximum value at a temperature of 365 °C.

In general, the pyrolysis of the lignocellulosic material starts with decomposition of hemicellulose (200 to 260 °C), followed by the decomposition of cellulose (240 to 350 °C), and finally lignin (280 to 500 °C). The mass loss in the range of 240 to 400 °C was mainly due to the chain scission and depolymerization of the lignin and cellulose chains by breaking the C-C and C-O bonds within glicopyranose ring units evolving water, carbon monoxide (CO), and CO_2 (Macedo *et al.* 2008). The maximum heating temperature in this study was 230 °C, so no other products were produced during the fabrication of the bio-board. The weight loss rate of the derivatives slowed down when the temperature rose to 287 °C. Presumably, the hemicellulose was completely degraded and only some cellulose remained, while the lignin was just starting to degrade, hence the change in the weight loss rate. When the heating temperature exceeded 365 °C, the derived weight loss rate decreased rapidly, and the cellulose was essentially completely degraded. When the temperature was above 500 °C, the remaining lignin tended to degrade completely until about 21.0% of ash remained.

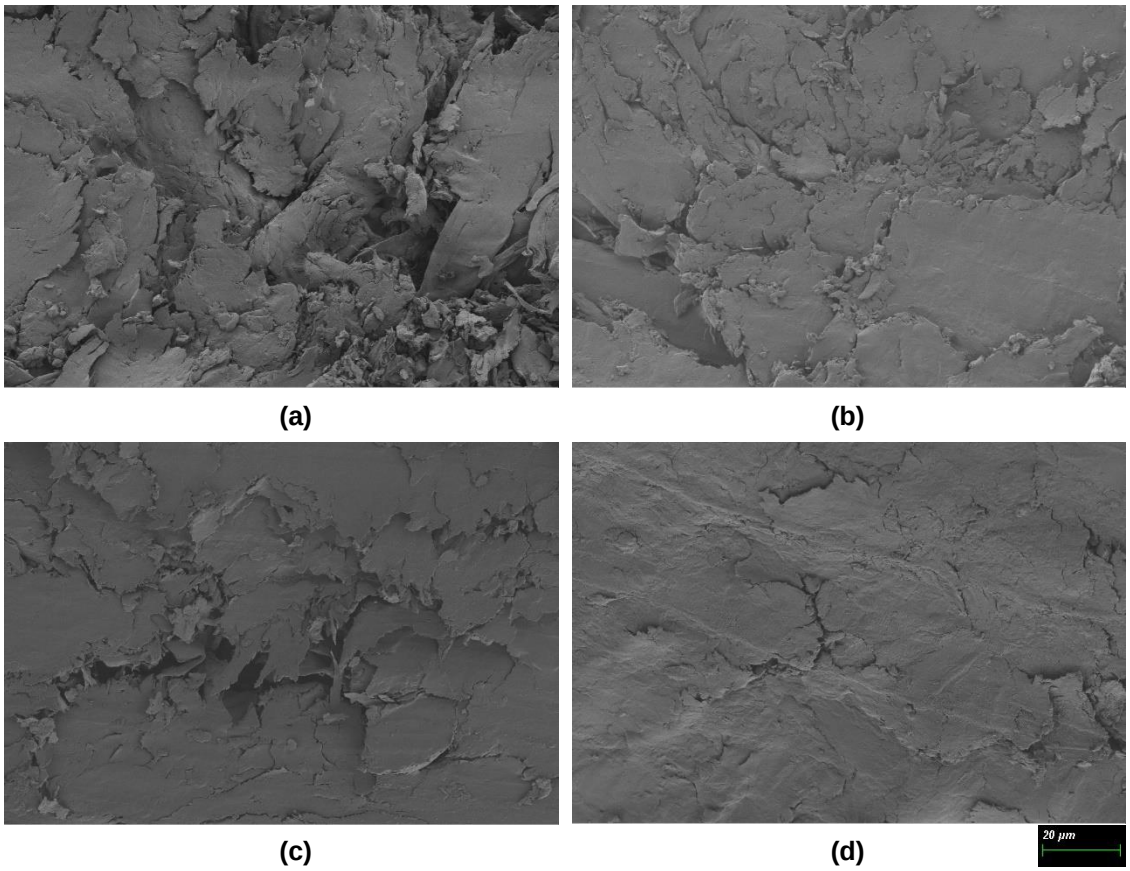

Fig. 2. Microscopic view of the bio-board (500× magnification). The bio-board samples produced at different applied pressures of a) 2.0 MPa and b) 8.0 MPa. The bio-board samples produced at different heating temperatures of c) 110 °C and d) 230 °C

Appearance of the Bio-board Samples (SEM Analysis)

The bio-boards were conditioned at constant humidity and room temperature 3 days prior to the measurements being carried out. In a macro perspective, considering that the bio-boards were binderless, the appearance of the boards did not have the phenomenon of falling slag, which indicates that the bonding between the wood fibers was relatively strong. The bio-boards were produced successfully under all the experimental conditions.

The microscopic morphology of the bio-boards is shown in Fig. 2. Irregular cracks were evident in the cross-sections of the bio-boards, which become smaller in length and width with increasing applied pressure and heating temperature. It is speculated that lignin may change from a glassy state to a highly elastic state at the glass transition temperature, whereupon it will be rubbery and ductile. Lignin acts both as a binder and it can fill the tiny voids, which contributes to the performance of the bio-board.

Analysis of the Functional Groups

The FTIR spectra of the produced bio-boards at different heating temperatures and applied pressures are shown in Fig. 3. In the functional group region, the peak at approximately 3330 cm^{-1} is due to the hydrogen bond O-H stretching. The hydrophilic tendency of the camphor tree was reflected in the absorbance band of 3000 to 3600 cm^{-1}, which is related to the -OH groups present in the aliphatic or aromatic alcohol and present in their main components (Bledzki *et al.* 2010). The presence of -OH bonds at approximately 3300 cm^{-1} was detected, while aliphatic C-H and methylene asymmetric C-H groups were observed at approximately 2900 and 2800 cm^{-1}, respectively (Sánchez *et al.* 2018). Between 1734 and 1595 cm^{-1}, two peaks appear, which could be caused by the benzene ring conjugating with the hydroxyl (-OH) or amino group ($-NH_2$), resulting in an absorbance peak due to ring structures (Figen *et al.* 2013). The moderate bands in the 1508 to 1156 cm^{-1} region is attributed to O-H bending and symmetrical and asymmetrical C-O-C stretching vibrations (Feng *et al.* 2018; Junior *et al.* 2018).

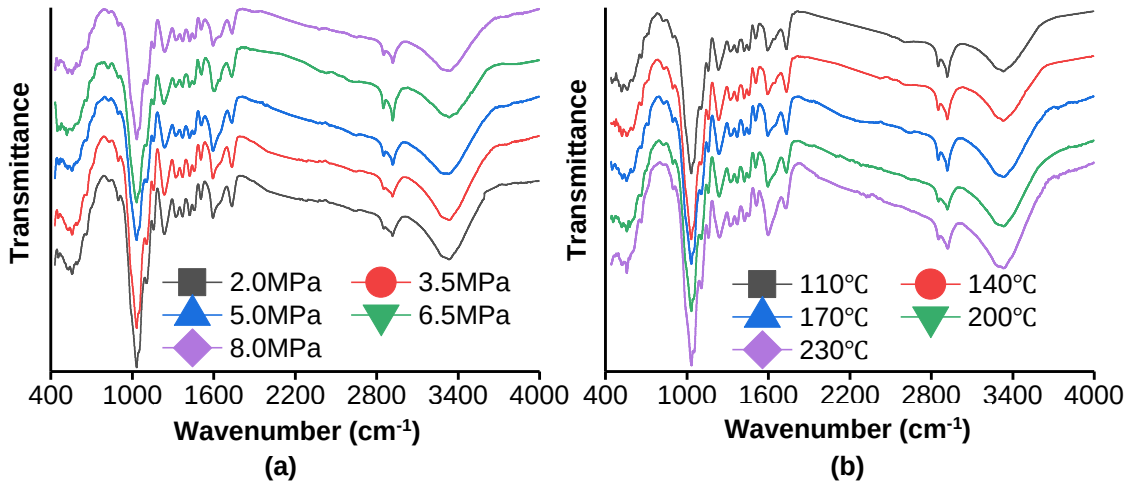

Fig. 3. FTIR spectra of the bio-board samples at a) different applied pressures and b) different heating temperatures

In the fingerprint region, the region of 1200 to 1000 cm^{-1} represents the C-O stretching and deformation bands in cellulose and lignin (Bledzki *et al.* 2010), which may be due to the high hydrophobicity of the bio-board samples. It can be noted that the

chemical bonding of the bio-boards produced under the two conditions were more similar, but the peak areas were different, which is related to their different contents of the same chemical bonds. In many cases, the peaks of the same components were shifted, which is related to the nature of hydrogen bonding and coupling effects.

Physical Characteristics of Bio-board Samples

Figure 4 shows the density and moisture content of bio-boards produced at the different experimental conditions. As shown in Fig. 4a, the densities of the bio-boards ranged from 0.900 to 1.080 g/cm^3 as the applied pressure increased. At the same time, the moisture content of the bio-boards produced in this experimental condition were in the range of 7.72% to 6.61%. As the heating temperature increased, as shown in Fig. 4b, the density of the bio-board also increased, with a minimum density of 1.029 g/cm^3 and a maximum density of 1.129 g/cm^3. In terms of the moisture content, a maximum of 9.91% was achieved at a heating temperature of 110 °C. The increase in the heating temperature resulted in a uniform and slow decrease in the moisture content, with a minimum of 3.66%. The density of the bio-boards produced in both cases exceeded 0.80 g/cm^3, which classified them as hardboards according to the JIS A5908 standard (2014). The differences in the effects of the applied pressure and the heating temperature on the density of the bio-board samples were relatively small, but the effect of changing the heating temperature on the moisture content of the bio-board samples was particularly noticeable.

As can be seen in Fig. 4, the densities of the bio-board samples were inversely related to the moisture content. The SEM images showed many tiny voids on the surface and inside the bio-boards, which are expected to accommodate a large amount of air and water molecules. The increased applied pressure resulted in tighter bonding between the fibers, which reduces the number of tiny voids and expels air and water molecules from the voids. The increased heating temperature intensified the evaporation of water, which caused the cellulose molecules to shed the water molecules bound to them. At the same time, the heating temperature facilitates the filling of tiny voids and the extrusion of water from the lignin in its highly elastic state.

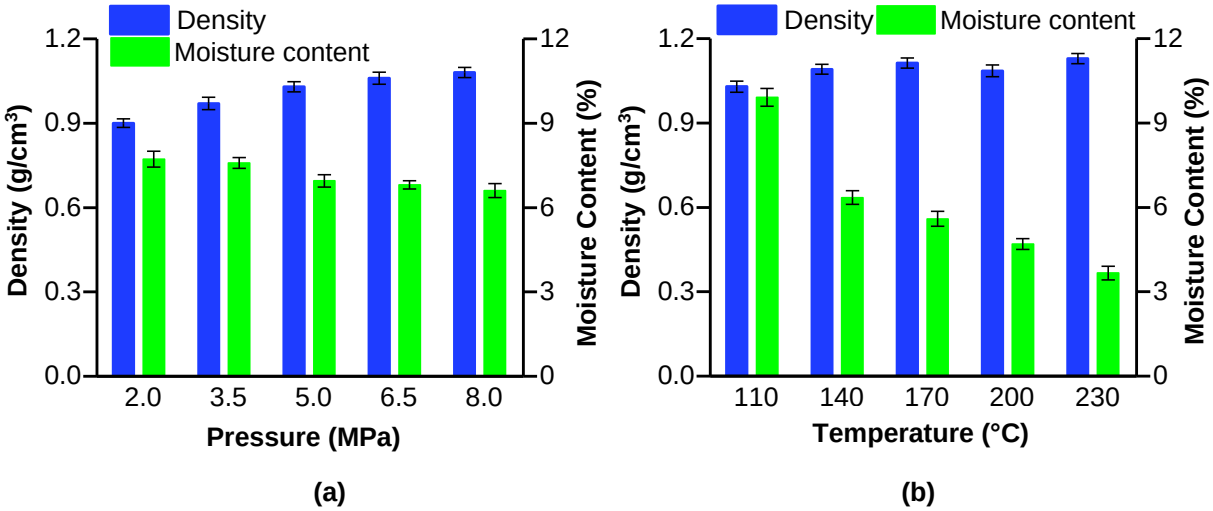

Fig. 4. The density and moisture content graphs of the bio-board samples at a) different applied pressures and b) different heating temperatures ($p > 0.05$)

Mechanical Properties

Figure 5 shows the tensile rupture stress and the bending rupture stress of the bio-boards produced at different experimental conditions. As shown in Fig. 5a, the bending and tensile rupture stress values increased as the applied pressure increased. The bending rupture stress increased from 15.44 to 28.20 MPa, and the tensile rupture stress increased from 9.80 MPa to 12.54 MPa. According to the physical characteristics of the different applied pressure discussed above, a decrease in the moisture content of the bio-board and a uniform increase in the density is shown in Fig. 4a. The reduction in the moisture content of the bio-boards indicates that as the applied pressure increased, the chemical bonds between the fiber molecules and the adjacent water molecules broke. This caused the fiber molecules near one another to be reconnected by hydrogen bonds and hydroxyl groups. This resulted in the detachment of water molecules from the bio-boards and a reduction in the distance between the fiber molecules, which facilitated tight bonding and mechanical entanglement between the fibers, forming covalent bonds. Therefore, the inter-fiber bonds tightened and the intermolecular van der Waals forces were enhanced, which improved the mechanical properties of the bio-boards.

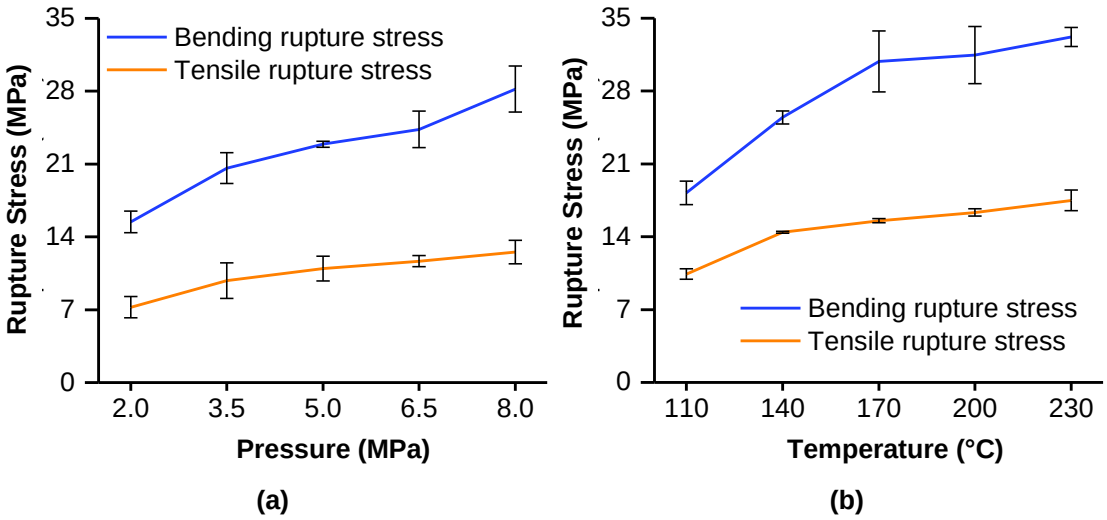

Fig. 5. The rupture stress of bio-board samples at a) different applied pressures and b) different heating temperatures ($p < 0.05$)

Similarly bending the rupture stress and tensile rupture stress values also increased as the heating temperature increased (Fig. 5b). The maximum bending rupture stress (33.2 MPa) and the maximum tensile rupture stress (17.49 MPa) were measured in the bio-board when heating temperature was 230 °C. The minimum bending rupture stress (18.21 MPa) and the minimum tensile rupture stress (10.40 MPa) were measured in the bio-board when heating temperature was 110°C. The reason for this phenomenon may be the reduced moisture content and softened lignin, which made the lignin more tightly bound to the fibers. Since the glass transition temperature of lignin is directly related to the moisture content (Jakes *et al.* 2019), when the heating temperature was above 170 °C, the mechanical properties of the bio-boards improved slowly, probably because the decreased moisture content of the bio-boards raised the glass transition temperature of the lignin. This created difficulties in the transformation of the lignin into the high elastic state and the binder content decreased. The softened lignin is beneficial as a binder to improve the

bending rupture stress and tensile rupture stress of the bio-board. Except for the bio-board sample made at 2 MPa in experimental condition A and at 110 °C in experimental condition B, the bio-boards that were produced at the remaining conditions satisfied the JIS A5908 standard (2014) for hardboard S20 type, which requires a bending rupture stress of 20 MPa. Overall, the bio-boards that were produced under different heating temperatures were superior to different applied pressures in terms of rupture stress.

Analysis of the Contact Angle

Wood is a hydrophilic material with a slight contact angle (Stark and Matuana 2007). The bio-board samples in this study were prepared using camphor wood residues with the contact angles shown in Fig. 6. The contact angle of all the bio-boards produced under two experimental conditions exceeded 90°. However, as the applied pressure increased, the contact angle increased from 111.85° to 116.36° at a gradual pace. The contact angle increased from 115.09° to 123.54° as the heating temperature increased. Although growth in contact angle relative to the temperature increase was slow, it was more pronounced than the applied pressure trend. The results showed that the bio-boards produced in this study had low wettability. In addition, the contact angle of the bio-boards under different heating temperatures were generally greater than the value of the contact angle under different applied pressures. Therefore, the heating temperature had a higher effect on the wettability stability aspects of the bio-boards than the applied pressure.

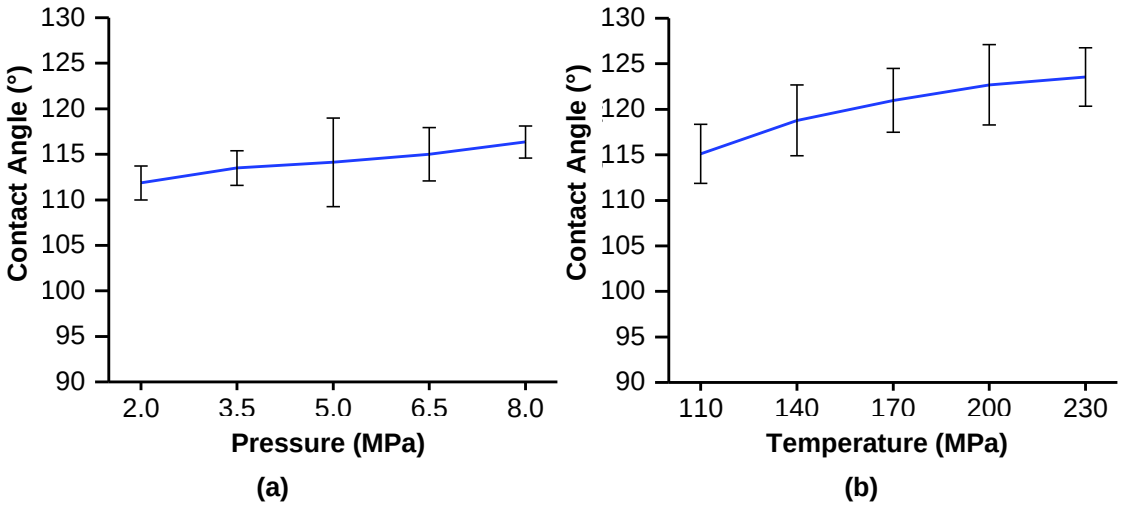

Fig. 6. The contact angle of the bio-board samples at a) different applied pressures and b) different heating temperatures ($p > 0.05$)

Dimensional Stability Properties

The results of the WA, TS, and LE testing on the bio-boards made according to the different experimental conditions are shown in Fig. 7. As can be seen in Fig. 7a, as the applied pressure was increased, the WA of the bio-boards decreased slowly from 101.11% to 94.4%, but the TS showed an upward trend from 50.7% to 76.6%. The LE value experienced a minimal change, increasing only from 2.50% to 3.12%. In Fig. 7b, as the heating temperature increased, the WA of the bio-boards rapidly decreased from 94.5% to 25.3%. There was also a large degree of change in the TS value from 77.9% to 20.6%. In terms of the LE value, the degree of change was relatively small, decreasing only from 3.63% to 1.28%. According to the JIS A5908 standard (2014), the WA of hardboard S20

should not surpass 30%. In this study, only the bio-board that was made at 230 °C met the JIS A5908 hardboard S20 type requirements for the WA value.

This phenomenon can be attributed to several things. Under different applied pressures, it may be due to the tight bonding and mechanical entanglement of lignin-rich fibers and the formation of covalent bonds (Sarkanen and Ludwig 1971). Concurrently, the fiber molecules were linked to hydroxyl groups by hydrogen bonds, which made them even less likely to bind water molecules. Meanwhile, the tiny voids on the surface of the bio-board became smaller, which prevented water molecules from getting inside the bio-boards as was confirmed by the SEM analysis. As the pressure increased, water molecules entered the interior of the bio-boards through many tiny voids and attached themselves to these tiny voids. In terms of the TS properties, the water molecules caused the tiny voids to swell rapidly. On the other hand, the fiber molecules combined with a layer of water molecules on the periphery, which thickened the fiber molecules and caused the thickness of the bio-boards to swell. In terms of the LE properties, the adsorption of the water molecules to fiber molecules did not cause them to elongate. Therefore, the small increase in the elongation was mainly caused by the expansion of the tiny voids.

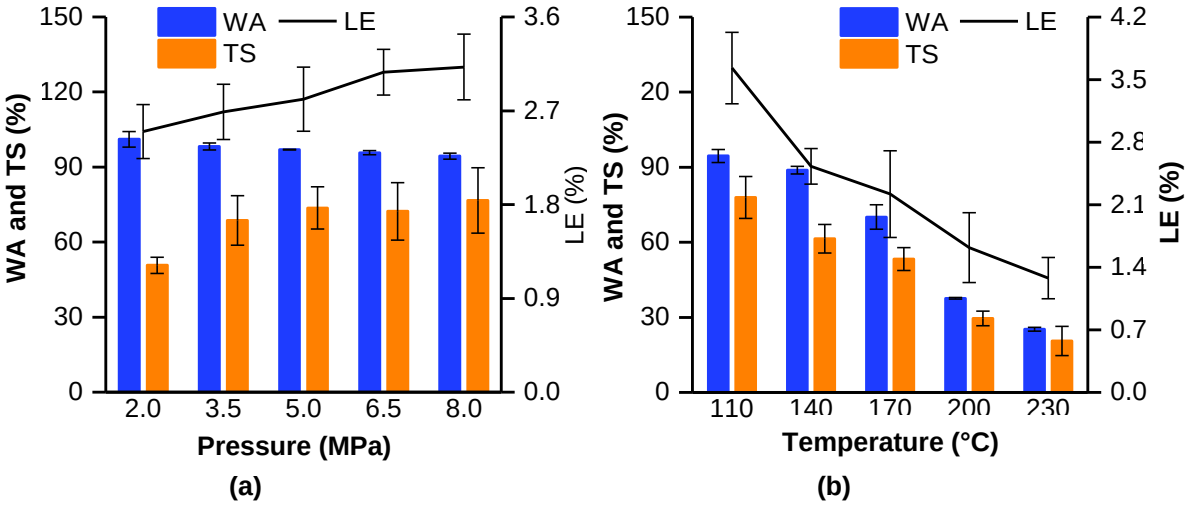

Fig. 7. The WA, TS, and LE values of the bio-board samples produced at a) different applied pressures and b) different heating temperatures ($p < 0.05$)

The following chemical reactions may be expected to occur under different heating temperatures. Owing to the poor thermal stability of hemicellulose, pyrolysis would first appear with the increased heating temperature. Numerous free hydroxyl groups in hemicelluloses, which are responsible for the wood wetting phenomenon, decreased strongly due to the degradation of the hemicelluloses. Moreover, hemicellulose is a hydrophilic substance, and its reduction would improve the waterproof properties to some extent (Özgenç *et al.* 2017). Another consideration is the condensation reaction of lignin. When the heating temperature exceeds 170 °C, the water absorption rate decreases rapidly. The TGA confirmed that the condensation reaction of lignin occurred from 200 °C, and the substance produced by the condensation reaction was black and hardly soluble in water, so the moisture content was significantly reduced after the water-soaking test (Feng *et al.* 2018; Song *et al.* 2020).

CONCLUSIONS

1. This study successfully produced new biodegradable bio-boards from the branch wood of camphor trees under different conditions without relying on chemical binders.
2. Both the applied pressure and the increase in heating temperature improved the properties of the bio-board, but the heating temperature had a higher effect on the bio-board than the applied pressure. The best performance of the produced bio-board was achieved when the fabrication conditions were 8 MP, 230 °C and 0.5 h.
3. Although the dimensional stability was improved to a certain extent under both experimental conditions, there were still deficiencies. In addition, the properties of the bio-boards did not continue to increase as the heating temperature and the applied pressure increased.

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