Ph.D. Thesis

Development of biodegradable biomass board and its properties using soybean straw

(大豆ガラを用いた生分解可能なバイオマスボードの開発研究)

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Preface

Conventional fiberboard is mostly composed of wood cellulose combined with petroleum-based resins. In recent years, the vast consumption of wood causes tremendous pressure on the supply of timber and influences global forest resources. Besides, it was inevitable that toxic formaldehyde gas was released during the utilization of fiberboard, which is harmful to the health of the human body and unfriendly to the environment. Given this, it is worthwhile to use other annually grown plant biomass to produce non-toxic, biodegradable fiberboard. The purpose of this research is to manufacture biodegradable biomass board (bio-board) by using soybean straw without any synthesis resin.

The procedure of manufacturing bio-board included cutting, soaking, refining, and forming. The manufacturing parameters, including applied pressure (2-8MPa), heating temperature (110-230 ℃), forming time (0.5-2.5 h), was evaluated, respectively. Two pieces of bio-boards were produced at each experimental condition. After that, the physic-mechanical properties of bio-board was evaluated by standard methods based on JIS A5905. The physical properties of the bio-board, including density and moisture content, were investigated. The mechanical properties of the bio-board, including bending strength, tensile strength, screw-holding force, and modulus of elasticity, were investigated. Additionally, the dimensional stability performance, thickness swelling (TS), and water absorption (WA), were evaluated.

Bio-boards were manufactured successfully at all experimental condition. These bio-boards were classified as hardboard according to JIS standard because the density exceeded 0.8 g/cm^3. With the increase of applied pressure, the bending rupture stress was in the range of 32.3 to 40.6MPa, the tensile rupture stress of the bio-boards was between 15.73 to 22.57MPa. The WA of bio-boards ranged from 87.7% to 97.1%, and the TS ranged from 45.8% to 62.0%. Generally, mechanical properties of bio-board were closely related to the density and moisture content affected by the applied pressure, whereas, bio-board had poor water-proof properties compared to the standard. The bending rupture stress of soybean straw bio-board was slightly increased from 39.3 to 43.2 MPa when the forming time raised from 0.5 to 2.5 h. The tensile rupture stress of soybean straw bio-board varied from 17.6 to 24.7 MPa. The WA of soybean bio-boards ranged from 97.2% to 123.4%, and the TS ranged from 66.2% to 97.8%. Except for the bio-board made at 0.5 h that had relatively low strength and water-proof performance, the performance at other forming time condition did not have significant difference.

With the increase in temperature, there was a decrease in moisture content, the softening of lignin, and the pyrolysis of hemicellulose, which was beneficial to the improvement of the mechanical properties of the bio-board. However, the excessive heating temperature, especially at 230 °C, did not significantly promote improvement to most mechanical properties. On the other hand, the dimensional stability of the bio-board was greatly improved from 140 to 230 °C.

Furthermore, to evaluate the influence of fiber length on properties of bio-board, soybean straw fiber was prepared with the three categories of fiber lengths: long fiber, short fiber, and mixed fiber. The bending properties, screw holding force, dimensional stability, and water soaking properties of these bio-boards were further investigated. The mixed fiber bio-boards had shown conceivably better mechanical properties and dimensional properties than long and short fiber length bio-boards, due to its dense structure in which short fibers were stuffed among the interweaving of long fibers.

Finally, two methods were tried to improve the water-proof of the bio-board in this research. The bending properties of the hybrid with 10% of PLA was comparable with pure bio-board, but the water resistance properties were highly improved. With the increase of PLA mass percentage, the bending properties were unexpected decreased, but the water-resistance properties were much more improved. Coating method was a valid method. Since the coating bio-boards had shown conceivably better bending properties and water-resistance properties than pure bio-board.

Bio-board which was made in this study performs well in mechanical properties. In the making process, none of chemical adhesive and chemical compound was used. The research of bio-board not only beneficial for solving the problem that traditional fiberboard releases toxic gases, but also had a good advantage of the utilization of agricultural wastes. It was also conducive to the protection of forest resources.

Acknowledgement

Firstly, I would like to express my sincerest appreciation and gratitude to my supervisor Prof. Xiulun Wang for his valuable advice and assistance both on my research as well as three years' international student life in Japan. Without his acceptance in energy utilization engineering laboratory, I wouldn't have had the opportunity to pursue doctoral degree at Mie University. His preciseness and pragmatic academic spirit and attitude gave me great help and inspiration during the process of conducting experiments and revising the thesis.

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List of figures

List of tables

Contents

Chapter 1. Introduction

1.1 Background and significance

The traditional fiberboard mainly uses wood as raw materials and adds different kinds of synthetic resin binders. Due to the reduction of wood resources in the world and the release of toxic formaldehyde gases in the use of fiberboard. In this case, it is significantly meaningful to utilize other annually growing plants biomass to produce non-toxic, biodegradable fiberboard.

1.1.1 Yield of agriculture straw resources

The five major food crops are the main root of agriculture straw resources, and the world's food crop area accounts for about the total crop area 85%. According to the report published by the Food and Agriculture Organization of the United Nations (FAO) (2019), global food production reached a record of 2.323 billion tons in 2019. Table 1 lists the yield of the four major food crops[1]. Corn production ranks first, followed by wheat production, and rice production ranks third.

Table 1 Grain production of main food crops in the whole world.

Year	Corn (million tons)	Rice (million tons)	Wheat (million tons)	Soybean (million tons)
2011	886.68	726.38	696.90	261.60
2012	875.04	736.60	673.73	241.34
2013	1016.21	742.50	710.40	277.67
2014	1039.23	742.45	728.73	306.35
2015	1052.14	745.91	741.70	323.31
2016	1127.04	751.89	749.19	335.61
2017	1164.47	769.83	773.88	353.03
2018	1147.69	782.00	735.18	348.71

Grass Valley ratio (The ratio between the amount of occurrence of crop straw and crop yields). Bi et al. [2][3] claimed that the Grass Valley ratio of wheat is 1.1, corn of 1.2, rice of 0.9, and soybean of 1.6. Based on this, Table 2 calculated the world's main straw yield. With the increase of agricultural products, as the by-product, a massive amount of agricultural straw generated annually. Only a small part of the crop straw was used in agricultural and industries application, e.g., as livestock feed and biofertilizer, as raw materials for pulp and papermaking fields. Most of the straw was discarded or burned, which not only polluted the environment but also wasted the biomass resources.

Table 2 Straw production of main food crops in the whole world.

Year	Corn straw (million tons)	Rice straw (million tons)	Wheat straw (million tons)	Soybean straw (million tons)
2011	1064.02	653.74	766.59	418.56
2012	1050.05	662.94	741.10	386.14
2013	1219.45	668.25	781.44	444.27
2014	1247.08	668.21	801.60	490.16
2015	1262.57	671.32	815.87	517.30
2016	1352.45	676.70	824.11	536.98
2017	1397.36	692.85	851.27	564.85
2018	1377.23	703.80	808.70	557.94

1.1.2 Physical and chemical properties of soybean straw

Soybeans (Glycine max (Linn.) Merr.) is an erect branching plant on average 3 to 5 feet tall. It has been derived from Glycine ussuriensis, which is a wild species growing in eastern Asia. Soy beans have been cultivated in China for at least 30 centuries. It was introduced into Japan and subsequently into Europe and North America, where it is extensively cultivated today.

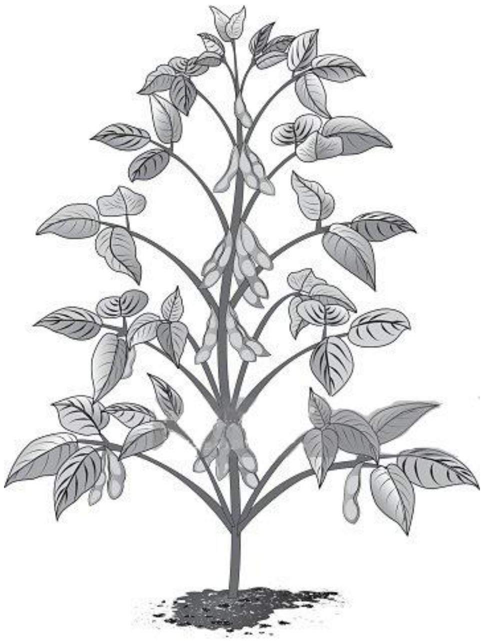


Fig.1-1 The products of soybean straw

Soybeans contain significant amounts of phytic acid, dietary minerals and B vitamins [4][5]. Traditional unfermented food uses of soybeans include soy milk, from which tofu and tofu skin are made. Fermented soy foods include soy sauce, fermented bean paste, nattō, and tempeh.

Soybean straw mainly consists of stem, pod, and root. The Fig.1-2 comes from the research of Liu et al., the stem is mainly constructed of a ground tissue system, a vascular tissue system, and a dermal tissue system. The ground tissue system in the stem is represented by the pith and cortex. The pith, which is composed of soft and light-colored spongy parenchyma cells, is in the center part of the stem. These parenchyma cells have simple, thin primary walls, and they comprise the "filler" tissue in the stem. They are also responsible for storing and transporting nutrients throughout the plant. According to the high-resolution images of the parenchyma, there were many pits on the cell wall. The cortex bounded on the outside by the epidermis is mainly composed of collenchyma cells that have irregularly thickened cell walls. Between the cortex and pith, there is the vascular tissue. It consists of two conducting tissues - the xylem and the phloem. The cells in this part have small diameters and thick cell walls. Some vessels that are distributed in this portion can transport water and nutrients from the roots throughout the plant.

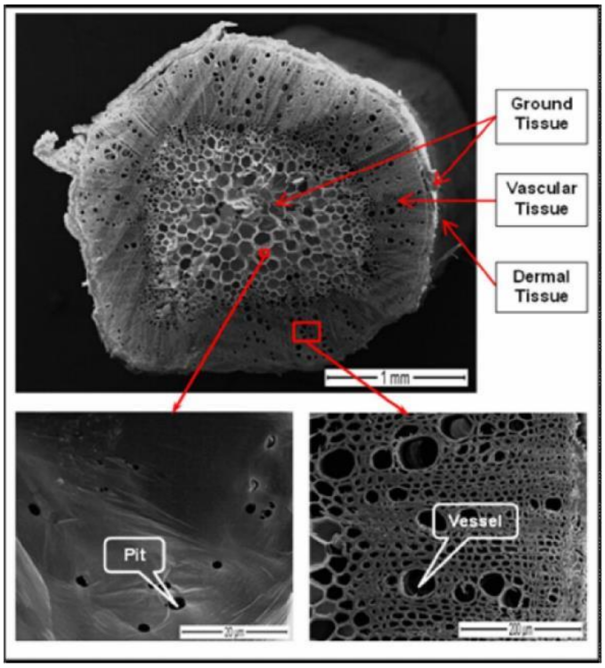


Fig.1-2 Microscope of soybean stalk in transverse section[6]

Raw soybean straw was composed of cellulose, hemicellulose, lignin, crude protein, ash, wax, and some other non-classified composition. The chemical component is different because the type of soy, place of origin, and analysis method are inconsistent. In general, the content of cellulose and lignin accounts for the main components, followed by hemicellulose[5].

Cellulose [$(C_6H_{10}O_5)_n$] is a linear polymer compound in which β-D-glucopyranosyl groups are linked by a 1→4 β glycoside linkage. Cellulose is the main component of plant cell walls, and which is insoluble in water and general organic solvents. Cellulose is the most widely distributed and most abundant polysaccharide in nature, accounting for more than 50% of the plant's carbon content.

Hemicellulose is a heteromultimeric composed of several different types of monosaccharides, including five-carbon sugars and six-carbon sugars. Specifically, hemicellulose contains xylose, arabinose, and galactose. Hemicellulose has hydrophilic properties and more readily hydrolyzed compared to cellulose.

Lignin is an exceptionally complex, water-insoluble polymer that does not have a definite primary structure and is formed by polymerization of different aromatic alcohols. It provides water-proofing, structural reinforcement and resilience with plant cell walls, giving their resistance to the biological and physical attacks.

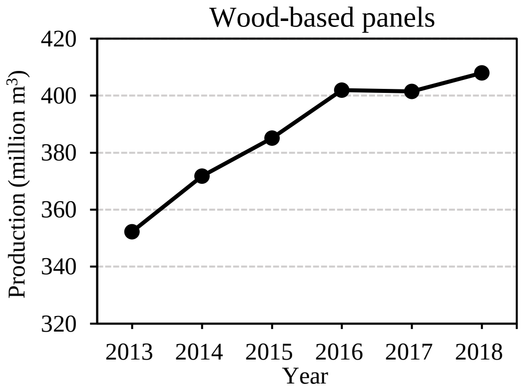
1.2 The development of non-resin fiberboard

The agriculture straw is widely available, inexpensive, and annually renewable. However, because of the lack of a comprehensive regulatory policy to manage straw resources, most of the straw is discarded or burned. These not only cause severe air pollution but also waste biomass resources. Therefore, it is an urgent issue to reduce the incineration loss of biomass resources and realize the comprehensive utilization of agricultural residues.

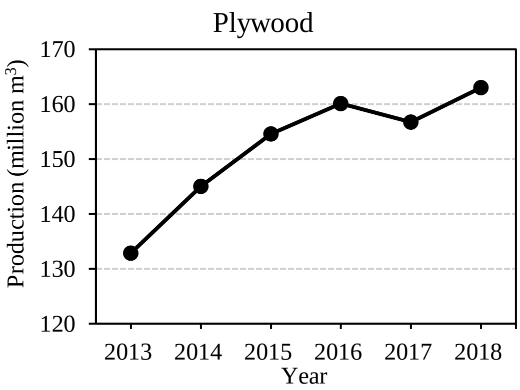
Biomass-based panels are an essential component of the use of biomass resources. The use of artificial board has greatly increased because of infrastructure needs in developing countries in recent years. Consequently, the vast consumption of wood causes tremendous pressure on the supply of timber, and influences global forest resources. Therefore, many kinds of composite materials have been developed using biomass straw as raw materials, including biomass straw fiberboard, biomass straw particleboard, biomass straw plywood, and biomass straw-degradable plastic composite board.

1.2.1 Production of artificial board

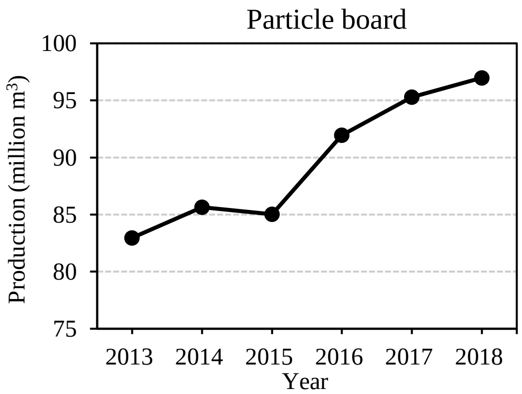
Wood-based panels are the dominant industry regarded in the utilization of biomass resources. The wood-based panels category is an aggregate category comprising plywood, particle board, OSB and fiberboard. According to the Forest Products stated from FAO Statistics (Food and Agricultural Organization of the United Nation), the global production of wood-based panels has grown steadily, reaching a total amount of 408 million m^3 in 2018. The details of each category were as follows: the plywood of 163 million m^3, accounts for 40% of total amount of wood-based panels; the particleboard of 97 million m^3, accounts for 23.77%; the fiberboard of 116.5 million m^3, accounts for 28.55%. After 2016, the fiberboard industry was affected by China's overcapacity and supply-side reforms, the production of fiberboard declined slightly.



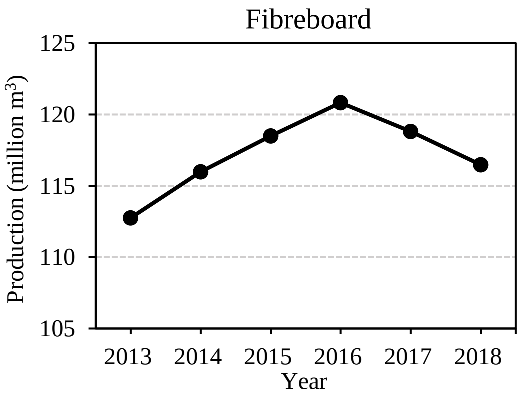
a. Production of artificial board in the world.



b. Production of plywood in the world.



c. Production of particle board in the world.



d. Production of fiberboard in the world.

Fig.1-3 Production of artificial board around the world in recent years.

Wood is the most important source of biomass raw materials to produce wood-based panels. In recent years, Due to increasing wood price and shortage of wood supply, there have been a large number of studies on the production of wood-based panels using agricultural straws and other annual plants. Agricultural straws [7] including rice straw, bagasse, corn straw, wheat straws, rape stalk, sorghum straw and soybean stalk have been explored due to their low price and richness. Some annual terrestrial plants and hydrophyte were also used to make panels, including Miscanthus sinensis, oil palm trunk, kenaf, Arundo Donax L., algae [17]. The use of non-woody raw materials can effectively alleviate the tight supply of wood, protect forest reserves, and greatly reduce waste of biomass resources, which is beneficial to protecting the environment.

The formaldehyde based synthetic adhesives are conventionally used by the wood composite industry. Urea formaldehyde (UF), melamine urea formaldehyde (MUF) and phenol formaldehyde (PF) are common types of commercial binders used in the manufacturing of MDF/HDF panels[22].

Due to the use of aldehyde additives in the process of manufacturing wood-based panels, it was inevitable that toxic formaldehyde gas was released. Formaldehyde has been identified as a carcinogenic and teratogenic substance by the World Health Organization. It was found that formaldehyde could cause genetic mutations in mammalian cells, and long-term exposure to formaldehyde increased the risk of malignant diseases such as cancer and leukemia. As a kind of widely used building decoration material, the release of formaldehyde from wood-based panels has caused widespread concern, and it is urgent to reduce the impairment of formaldehyde to human being and environment.

1.2.2 The development of non-resin fiber board

The release of toxic formaldehyde gas during the application of fiberboard in daily life, has caused environmental and health concerns. Binderless board is beneficial, being manufactured by relying on the self-bonding of the internal composition of raw materials instead of using synthetic binders to solve this problem.

Two different method has been used in the production of fiberboard, i.e., dry-forming process and wet-forming process. The dry-forming process uses air as the fiber carrier. The fiber is prepared by a single separation method. Generally, it does not need to finely ground, and the synthetic resins are mixed with fiber to improve the bonding ability. After the distribution of mixture into a mat, it undergoes pre-press and hot pressing to finally produce fiberboards. The wet-forming process uses water as the carrier. The wet-forming process involves the distribution of fiber into water. Hydrogen bonds formation and thermosetting adhesive behavior of lignin are expected during heating and drying processes. Accordingly, less, or none binders are needed. Therefore, the wet-forming process is a more effective method for the production of non-resin bio-board.

It was reported by Hubbe that the inter-fiber bonds such as hydrogen bonding between fibers and dispersion force were the main factors influencing the properties of panels rather than individual fiber strength[23]. Chemical pretreatment has been applied for facilitating the adhesive ability among fibers. Alkaline [24], acid [25], and oxidation agents [26] are commonly used as chemical agent. Halvarsson et al. [27]made fiberboard by using wheat straw fiber that was treated through Fenton's reagent. The result showed that the mechanical properties were enhanced with the increase of hydrogen peroxide. Li et al. [28] evaluated some mechanical and physical properties of MDF (Medium Density Fiberboard) whose raw materials were pretreated with oxalic acid. While this treatment increased the water resistance to a certain extent and was beneficial for the lightness of board, there was no promotion for properties of internal bond strength of the panel. Enzymatic pretreatments were also used for promoting fiber bonding [29]. Laccase is a kind of polyphenol oxidase containing copper, which is found in fungi, higher plants, bacteria, and some insects [30]. It was reported by Nasir et al. [31], who used SEM to observe a fiber surface, which was pretreated by laccase. The morphology manifested a smooth fiber surface covered with a uniform layer of softened lignin, which enhanced the fiber bonding ability. Whether it was chemical pretreatment or enzymatic pretreatment, reaction time, and reagent concentration shall be controlled to avoid excessive removal of lignin and decomposition of cellulose. At the same time, these two methods have a limited effect on improving dimensional stability.

Lignin holds a large proportion of vascular plants and some algae. It acts as a binder and reinforcement in the cell wall formation [32]. Therefore, the addition of lignin and full use of lignin as an intermediary for fiber bonding is expected to enhance the properties of non-resin board. Anglès et al. [33] evaluated the effect of different components of lignin on the properties of the board, which proved that water stability and mechanical properties were noticeably increased. Domínguez-Robles [34] also explored the different percentage of lignin content on mechanical properties of wheat straw board, and results showed that the mechanical properties were proportional to the lignin content (from 0% to 15%). Furthermore, Zhou et al. [35] manufactured binderless fiberboard by using cotton stalk with enzymatic hydrolysis lignin, and physico-mechanical property tests were carried out. It turned out that the self-bond ability among fibers increased with the softening of lignin, which was closely related to fiber moisture content and pressing temperature. Okuda et al. [36] produced binderless board using kenaf core, and explored the physical properties and chemical changes during the hot-press stage. The results showed both the softening of lignin and condensation reactions of lignin took place during this period. These changes were in favor of enhancing the fiber self-bonding ability.

1.3 Research objectives

The overall aim of this research was to manufacture bio-board (biodegradable fiberboard) by using soybean straws which consist of a large amount of lignin, with performance comparable to commercial standards. The main objectives of this experimental research are as follows:

- To investigate the effect of manufacturing parameters on the properties of bio-board;
- To evaluate the effect of fiber length on properties of bio-board;
- To improve the water-proof of bio-board.

The procedure of manufacturing bio-board, including cutting, soaking, refining, and forming, was proposed. The manufacturing parameters, including applied pressure, heating temperature, forming time, was evaluated. The physical properties of the bio-board, including density and moisture content, were investigated. The mechanical properties of the bio-board, including bending strength, tensile strength, screw-holding force, and modulus of elasticity, were investigated. Additionally, the dimensional stability performance, measured by water absorption (WA), thickness swelling (TS), and linear expansion (LE), was evaluated. For the effect of forming time, the properties of corn straw bio-board were also evaluated. For the effect of heating temperature, thermal gravimetric analysis, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) were used to investigate the thermal stability of raw materials, chemical changes, and microstructural changes corresponding to different heating temperatures.

To evaluate the influence of fiber length on properties of bio-board. Soybean straw fiber was prepared with the three categories of fiber lengths: long fiber, short fiber, and mixed fiber. Three different kinds of bio-boards were made, by using these three fibers. The bending properties, screw holding force, dimensional stability and water soaking properties of these bio-boards were further investigated.

Two methods were tried to improve the waterproof of the bio-board. First, the method of mixing the degradable PLA with soybean straw fibers was used. PLA is hydrophobic materials. The addition of a certain amount of PLA is expected to improve the waterproof of the bio-board. In this study, bio-boards with different mass percentages of PLA were prepared and their effects on water barrier and bending properties were investigated. Besides, a water-repellent coating was made on the surface of the bio-board. In this study, PLA was used as coating materials, and the effect of PLA on barrier and bending properties were evaluated.

Chapter 2. The manufacturing and characterization of soybean straw bio-board

2.1 Introduction

The hot-pressing method of the artificial board can be divided into the flat press method, rolling press method, and extrusion method according to its production method. The heating method consists of contact heating, high-frequency heating, and contact high frequency mixed heating. The operating conditions of hot-pressing equipment consist of continuous state and periodic state [37][38]. Periodic hot-pressing has a simple structure, in which the mat is heated and pressed in a fixed state. Generally, the flat pressing method is selected. Therefore, the periodic hot-pressing is suitable for small-scale production and testing. Continuous hot pressing with high product accuracy and efficiency is suitable to produce large-scale wood-based panels. Constant hot pressing is classified into continuous rolling pressing and continuous flat pressing. The former's structure is relatively simple with limited pre-curing of the product, and the secondary process of filming can be further performed. Nevertheless, the produced board has the problems of low static bending strength, sizeable internal stress, and being prone to warp deformation and large deviations in density and thickness. Therefore, it is generally used to produce panels with a thin dimension. Continuous flat pressing can eliminate warping deformation, precisely control the thickness of the board, and the product quality is excellent. Therefore, continuous large-scale production is more commonly utilized.

In the process of producing particleboard by the extrusion method, the blanks that fall between the two hot-pressed plates are pressed by the reciprocating punching heads up and down, and the resins is cured by heating the hot-pressed plates to form boards [39]. This method has some favorable properties of less amount of resin and high heat transfer efficiency. Besides, the produced board has a low water thickness swell and small thickness tolerance. However, the board made by using this method has a lower density and static bending strength. It is commonly used to produce hollow particleboard. The non-resin bio-board in this study is still in the experimental stage, so the flat hot-pressing method with the simplest structure was used.

2.2 Manufacturing of soybean straw bio-board

Dried soybean straw obtained from the experimental farm of Mie university was to make bio-board. The procedure of manufacturing bio-board, including cutting, soaking, refining, and forming, is shown in Fig. 2-1. The lower part of Fig. 2-1 is the transformation of materials from straw to fiber and finally turned into the bio-board.

Fig.2-1 The making process of bio-board.

First, the soybean straw was cut by an electric disintegrator (SU16, CO WA cutter Corp., Mishima, Shizuoka, Japan) into chips shorter than 10 mm. After that, the tiny chips were immersed in water at 20 °C for 96 h. In the process of soaking, the fibers were softened and swelled gradually. This process was beneficial for refining treatment, which converted soybean straw into pulp.

In the refining process, the soaked soybean straw was fibrillated by a beat refiner (Model A Beat finer, Satomi Corp., Shizuoka, Japan) as shown in Fig. 2-2. The fiberization of soybean straw was conducted by using a waterway circulation system of the refiner, in which the straw, along with running water, passed through rotating blades repeatedly. As shown in Fig.2-3, the coarse sieve was used to remove the long fiber chips. In the end, the soybean straw became tiny fibers, and the generated fiber pulp could pass through the sieve of 2 mm.

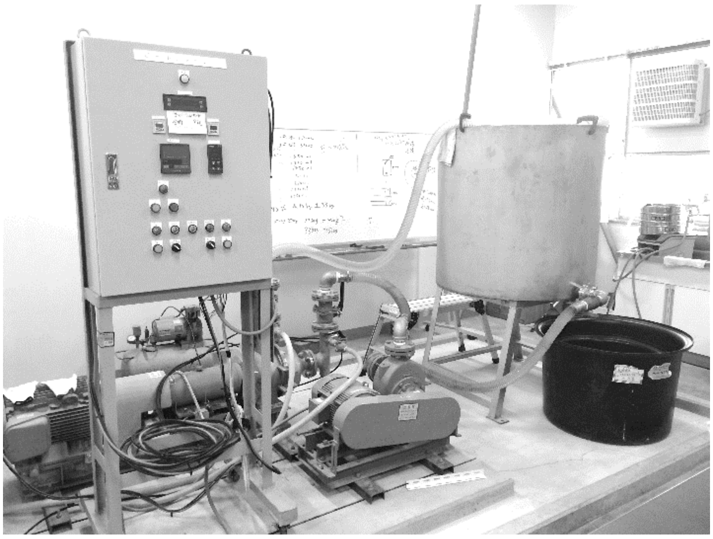
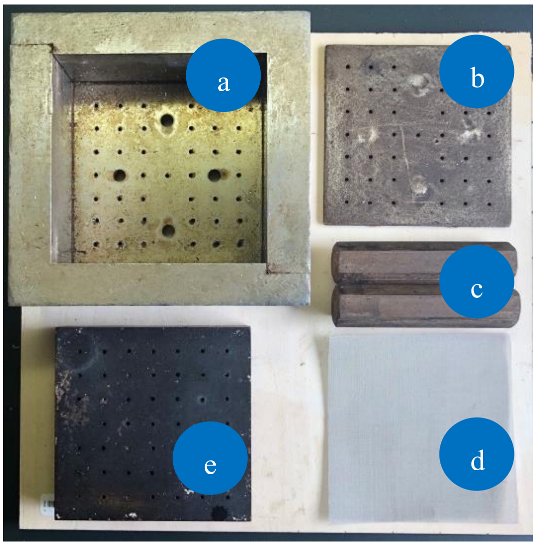


Fig.2-2 The electric beat refiner system.

Fig.2-3 Generated soybean straw pulps

Next, in the forming process, the bio-board mat was prepared by using the appliance as shown in Fig. 2-4. The stainless-steel mold with 100 mm in length, 100 mm in width and 40 mm in depth. Aluminum plate and stainless meshes with same dimension were placed into the bottom of mold successively. Then, the generated straw pulp was poured into the mold to form evenly distributed fiber mat. Finally, the stainless meshes and copper cover and two copper sticks was placed on the top of the fiber mat in turn. There were many 2 mm diameter holes distributed in the bottom of mold, copper cover and plate, in which the excessed water could squeezed out.



a) stainless steel mold; b) aluminum plate; c) copper sticks; d) stainless meshes; e) copper cover.

Fig.2-4 The appliance used in the forming process

The hot-press machine (IMC-180C Imoto Corp., Kyoto, Japan) as shown in Fig.2-5 was used to fabricate the bio-board. Firstly, the papered mold with bio-board mat was placed onto platform of the hot-press, and pre-pressing was carried out first at room temperature. Secondly, the pressure and heat applied to the metal mold was controlled by the hot-press machine with a manually controlled hydraulic press system. The heating temperature of hot-press was controlled by the PID method, and the maximum heating temperature was up to 340 °C. The applied pressure adjusted from 0 to 12.4 MPa. The produced bio-board were conditioned at a constant humidity and room temperature more than three days before the measurements and tests were conducted.

Fig.2-5 The hot press machine used in this study.

2.3 Characterization of soybean straw bio-board

2.3.1 Thermogravimetric analysis

The thermogravimetric analyses (TGA) of the raw materials were conducted using TA Q500 (TA Instruments, New Castle, DE, USA) thermogravimetric analyzer. Twenty milligrams of dried soybean straw fiber were placed into the alumina crucible of the TA Q500 and heated from 25 °C to 700 °C at a heating rate of 20 °C min^{-1} under a nitrogen atmosphere.

2.3.2 Spectroscopic analysis

FTIR spectroscopy was used to characterize the type of functional groups existing in samples from produced bio-boards. Sample pellets were mixed containing 5 mg of bio-board powder and 95 mg of finely ground potassium bromide (KBR), which was then pressed into pellets lower than 1 mm in thickness. After that, the prepared pellets were investigated with a Nicolet 380 FTIR spectrometer (Thermo Fisher Scientific Corp., Waltham, MA, USA) between 4000 cm^{-1} and 400 cm^{-1} with a resolution of 2 cm^{-1}.

2.3.3 Scanning electron microscopy analysis

SEM analysis was used to conduct the cross-section observation related to the fiber bonding quality inside bio-board. The SEM samples with a dimension of 0.5 cm × 0.5 cm were cut from bio-boards using an ultrasonic cutter (ZO-40B Honda Plus+ Corp., Shinshiro, Aichi, Japan), in which a smooth cross-section surface could be generated. Then, the samples were coated with gold using the field emission Inspect SEM (Inspect F50, FEI Corp., Hillsboro, OR, USA). Furthermore, the microstructure of bio-board was observed with FEI Inspect at 10.00 kV in Secondary Electron Imaging (SEI) mode.

2.3.4. The test for mechanical properties

The densities were measured for each bio-board by means of dividing the bio-board's mass to its volume. The volume is obtained by measuring the length, width, and height. The calculation methods for these dimensions are shown in the Fig.2-6. After that, the bio-boards were divided into four specimens for bending strength test and three specimens for tensile strength test, as illustrated in Fig. 2-7. Other bio-boards were divided into specimens for screw-holding force test and dimensional stability test. The above-mentioned mechanical properties and dimensional stability performance of bio-board were evaluated according to Japanese Industrial Standard JIS-A5908[40].

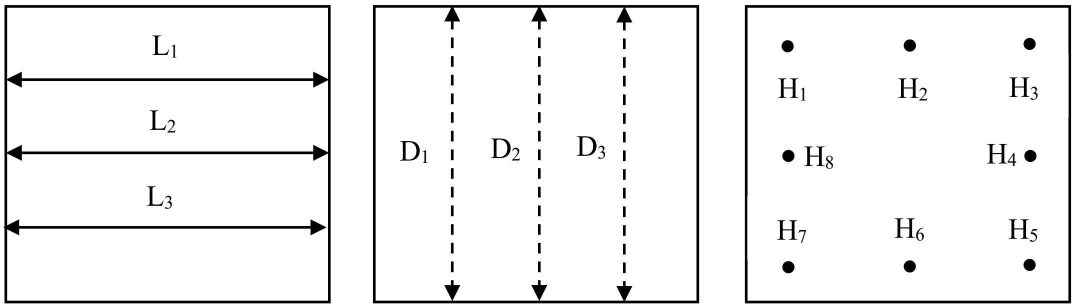


Fig.2-6 The calculation method of dimension of bio-board

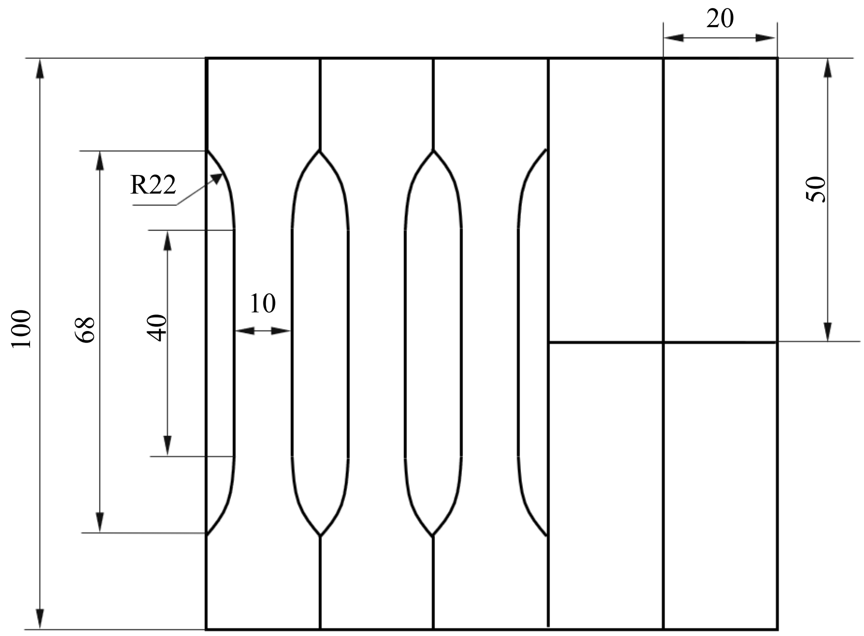


Fig.2-7 Specimens for bending test (50×20) and tensile test (100).

The mechanical properties tests were conducted with a universal material testing machine (SVZ-200NB-200R2, IMADA Corp., Japan) as shown in Fig.2-8. The instrument has automatic compensation function. Table 3 lists the main parameters of universal test machine. The measurable load was in the range from 20 to 2000 N. The resolution of load measurement was 0.1 N, and displacement measurement resolution was 0.01 mm.

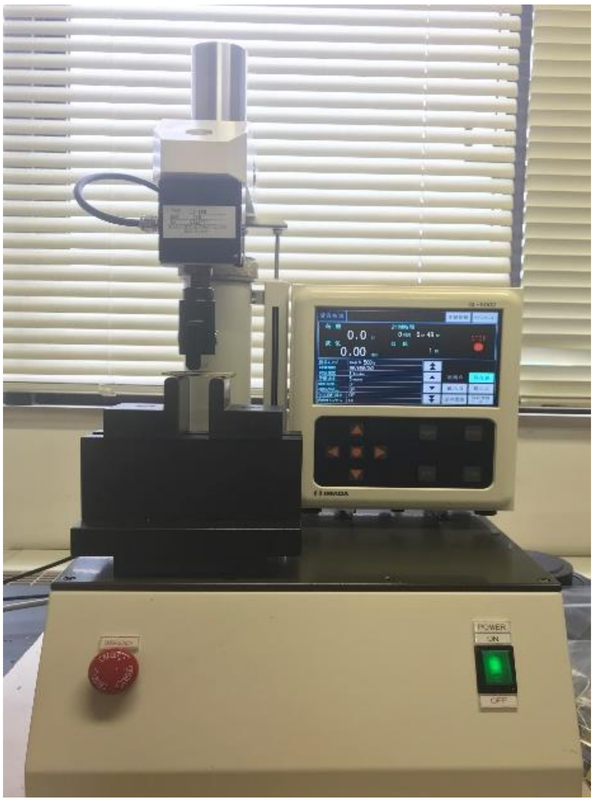


Fig.2-8 Universal test machine

Table 3 The main parameter of universal test machine

Model specification	SVZ-200NB
Maximum load	2kN
Load accuracy	Each measurement value within ± 1.0% (Test force 20 to 100%)
Display displacement	0.01～130.0 mm
Crosshead speed	5～500 mm/min
Stroke	130 mm
Sample mounting	197～295 mm
Distance to the	100 mm
Table size	100×150 T With grooves
External output	Dedicated USB
Power supply	AC100V 5A
Size	W338×D357×H642(mm)
Weight	About 53 kg
Safety device	Optional setting upper and lower limit device; Overload protection function

The dumbbell-shaped specimens were used for tensile test at a test speed of 10 mm/min. Tensile rupture stress was calculated by using Eq. (1). Bending rupture stress was obtained from specimens which had dimensions of 50 mm×20 mm. The specimens were loaded with a central loading nose and two lower supports, each having a radius of 5.0 mm, and the distance between the two lower supports adjusted and set to 40.0 mm. During the test, the load was applied with test speed of 10 mm/min. Bending rupture stress was calculated by using Eq. (2). Modulus of elasticity (MOE) is the ratio of bending stress and strain within the range of elastic deformation stage. As shown in Fig.4, the elastic deformation stage was chosen between 10% of σ_{max} and 40% of σ_{max} as suggested in ASTM D1037 in this study. the MOE was defined as the equation (2.2)

$$\sigma_b = \frac{3Wl}{2bh^2} \tag{1}$$

$$\sigma_t = \frac{W}{bh} \tag{2}$$

$$MOE = \frac{l^3\left(F_2 - F_1\right)}{4bh^3\left(a_2 - a_1\right)} \tag{3}$$

where σ_b is bending stress, σ_t is tensile stress, F is applied load, l is supported span, b is width of specimen, and h is thickness of specimen. F_2-F_1 is the increment of load on the straight-line portion of the load-deflection curve; a_2-a_1 is the increment of deflection at the mid-length of the test piece.

Fig.2-9 Three-point bending test and tensile test of bio-board.

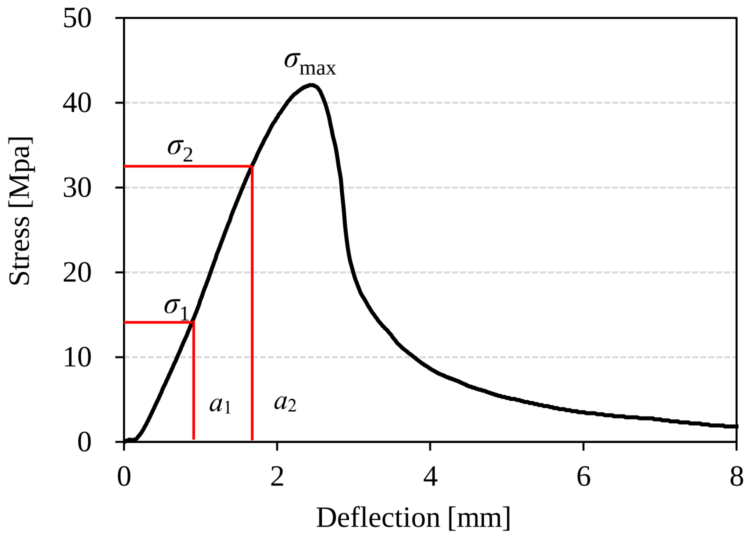


Fig.2-10 Stress-deflection curve of bio-board

After the bending test and the tensile test, the specimens of two tests were used to calculate the moisture content. Samples of different conditions were placed into aluminum boxes, separately. Then the moisture of specimens was evaporated in a dry oven (LDO-450S, IUCHI Corp., Tokushima, Japan) with a temperature of 110 °C for 24 h. Moisture content was calculated by using Equation (4).

$$MC = \frac{m_a - m_b}{m_a - m_c} \tag{4}$$

where MC is moisture content, m_a is mass of samples before evaporation, m_b is mass of samples after evaporation, and m_c is mass of empty aluminum box.

Fig.2-11 Dry oven and specimens for measuring the moisture content

According to the provisions of JIS-A 5908, the screw holding force test was carried out by using the screw with 2.7 mm in diameter, and the screwed depth was 11mm. Owing to the limitation of dimensions of mold, it was hard to make a bio-board with a thickness greater than 11 mm in this experiment. Based on the JIS-A 5908 standard, after trials and tries, when the thickness was less than 11 mm, the calculation formula of the screw holding force was summarized as Eq. (5)

$$SHF = \frac{CF_{\max}}{hd} \tag{5}$$

where $F_{\max}$ is the maximum load, h is thickness of specimen, d is the nominal diameter of wood screw, and C is correction factor compared to the standard experiment, which is 25.155.

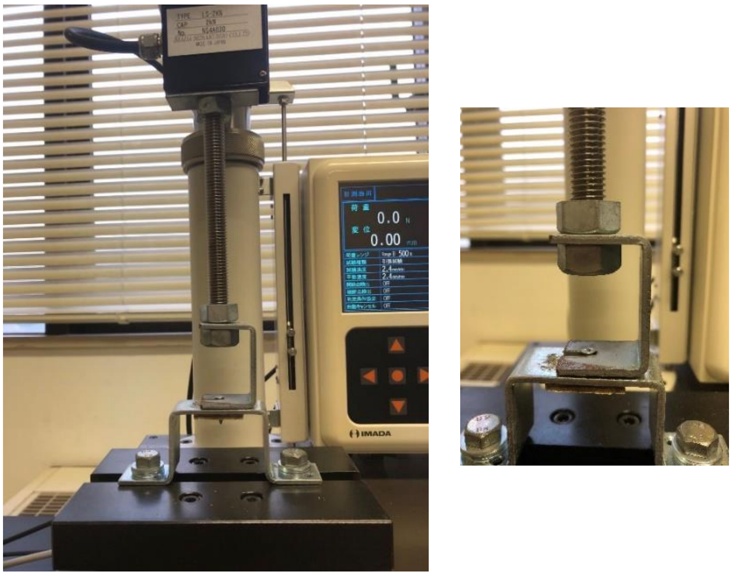


Fig.2-12 Screw holding force test of bio-board

2.3.5 The test for dimensional stability

Water absorption (WA), thickness swelling (TS), and linear expansion (LE) were measured to evaluate the water soaking and dimensional stability of bio-board. The 25 mm × 25 mm specimens were soaked in water at the depth of about 2 cm below the water surface, then placed in an incubator (CFH-415, Tomy Corp., Japan) with the temperature of 20 ± 1 ℃ for 24 hours. Thickness, length, and weight were measured before and immediately after water absorption test. These parameters calculated by the Equation. (6) - (8).

$$WA = \frac{m_2 - m_1}{m_1} \tag{6}$$

$$TS = \frac{t_2 - t_1}{t_1} \tag{7}$$

$$LE = \frac{l_2 - l_1}{l_1} \tag{8}$$

Where, WA is water absorption rate; m_1 is mass before immersion; m_2 is mass after immersion; TS is thickness swell; t_1 thickness before immersion; t_2 is thickness after immersion; *LE* is linear expansion; l_1 is length before immersion; l_2 is length after immersion.

Fig.2-13 Incubator and specimens used in water absorption test

2.3.6. Statistical analysis

All data reported were the average values of duplicates. Experiment data were evaluated by one-way analysis of variance (ANOVA), and $p < 0.05$ was considered statistically significant.

2.4 Summary

This chapter first summarizes the typical industrial fiberboard production process, then elaborate on the making process and technical details of the biomass board in this study, and finally discusses the characterization method used in this study to evaluate board properties.

Chapter 3. Effect of applied pressure on properties of bio-board

3.1 Making condition

The making condition such as heating temperature, applied pressure, and forming time undoubtedly influenced the properties of bio-board. This chapter mainly discusses the effect of applied pressure on properties of bio-board. First, 10 cups of approximately 500 ml soybean straw pulp were prepared by using measuring cylinder. The applied pressure was 2.0 to 8.0 MPa with an interval of 1.5 MPa. The condition of heating temperature was 110 °C, and forming time was 2 h. Bio-board was made under various conditions shown in table 2.

Table 4 The making condition of bio-board.

Item.	Heating Temperature (°C)	Applied pressure (MPa)	Forming time (h)
A	110	2.0	2
B	110	3.5	2
C	110	5.0	2
D	110	6.5	2
E	110	8.0	2

Finally, two pieces of bio-boards were produced at each forming condition. Fig.1. shows the produced bio-board at applied pressure of 5MPa.

Fig.3-1 Produced bio-board at applied pressure of 5MPa.

3.3 Results and discussion

3.3.1 Density and moisture content

In order to know the density of bio-board made in this experiment, the dimension and mass of bio-board were measured. The results of the mass and thickness of bio-board is shown in Table 2. The mechanical properties of the boards could be compared without considering the variation about initial difference in raw material quantity.

Table 5 The mass and thickness of bio-board made at different applied pressure

Item		Average	Standard deviation	Coefficient of variation (%)
Mass (g)		13.654	0.340	2.494
	A	1.534	0.136	8.855
	B	1.406	0.103	7.357
Thickness (mm)	C	1.401	0.127	9.081
	D	1.331	0.072	5.384
	E	1.262	0.094	7.415

The thickness of bio-board was also measured at eight points on each bio-board and the results of the thickness measurements recorded and displayed in Table 2. Overall, with the increases of applied pressure, the average thickness of the bio-board decreased. The coefficient of variation (CV) represents the ratio of the standard deviation to the average, and the CV of the thickness value for each bio-board is less than 15%. Therefore, the thickness of bio-board has good consistency, which has no significant influence on the mechanical properties of the bio-board.

The density of the bio-boards produced under various forming pressure are shown in

Fig. 3. When the pressure increased from 2 to 8 MPa, the density increased from 0.89 to 1.06 g/cm^3. These bio-boards are classified as hard board according to JIS standard because the density exceeded 0.8 g/cm^3. Fig. 4 indicates the moisture content of the bio-boards produced under various applied pressures. The moisture content fluctuated from 6.14% to 7.10% inversely proportional to the applied pressure, although the pressure has little effect on moisture content.

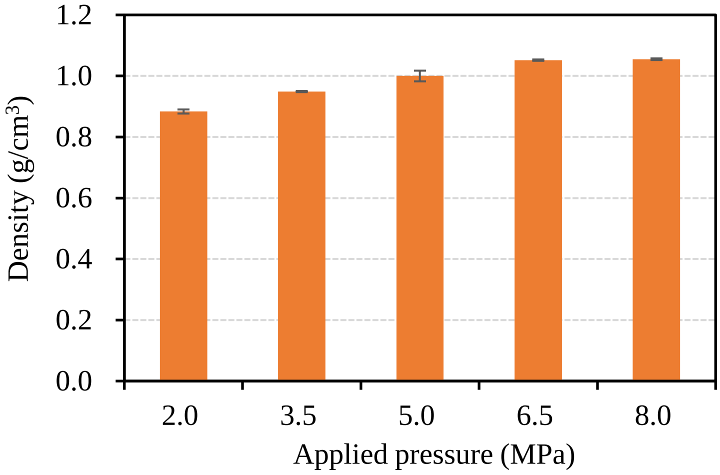


Fig.3-2 Density of bio-board at different applied pressure

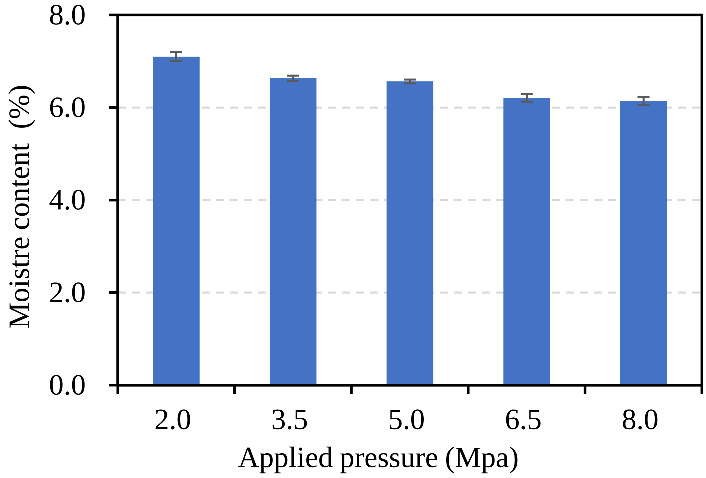


Fig.3-3 Moisture content of bio-board at different applied pressure

3.3.2 Mechanical properties of bio-board

As shown in Fig.3-4, the fracture of the tensile test varied from different specimens. Fracture failure occurs at different positions of samples, and the fractured cross-section is also not the same, in which some of the sections are inclined. It shows that the chemical bonding capacity among fibers is different. Therefore, the inside of the bio-board is not a homogeneous state.

During the bending test, the stress on the upper and lower surfaces of the specimens is different, the lower surface is subjected to tensile forces, and the upper surface is subjected to compressive forces. Therefore, the failure of the bio-board may include tensile fracture, compression failure, or both. The Fig.3-5 shows the typical fracture failure diagram of bending test specimens of the soybean straw bio-board. The tensile fracture causes the failure of the soybean straw bio-board. The tensile properties of biomass straws are generally worse than the compressive properties during the bending test.

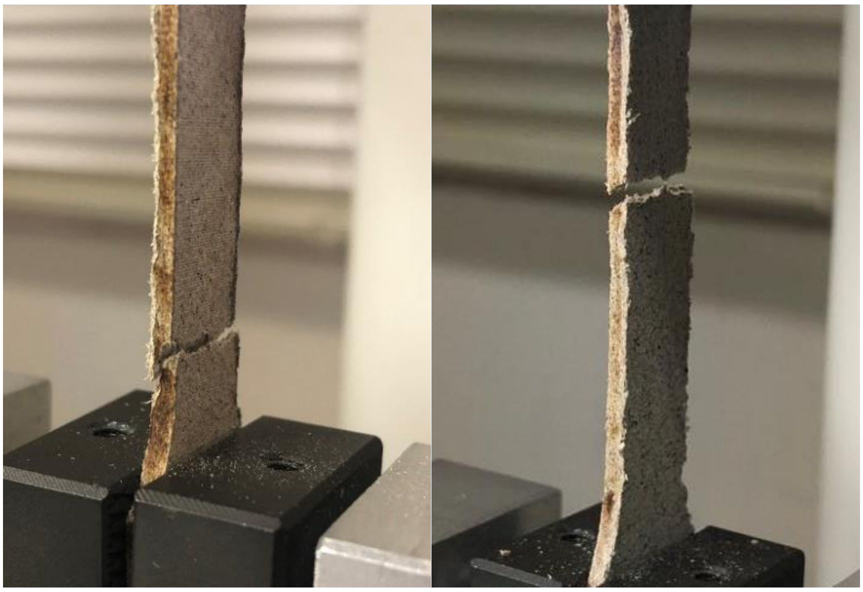


Fig.3-4 Fracture of tensile test of different specimens

Fig.3-5 Fracture of bending test of soybean straw bio-board specimens

As an example, Fig. 3-6 shows the tensile stress-strain curves of specimens from bio-board C, which were marked as Ct1, Ct2, and Ct3. During the initial period of stretching, the specimen materials were in the stage of elastic deformation. When the stress increased, the stage of elastic-plastic coexistence occurred. Finally, the failure happened and the stress reached the maximum value then the stress suddenly decreased to zero, the maximum value was defined as rupture stress. For these three specimens, the stress-strain curves did not coincide entirely because this specimen was not homogeneous. The stress-strain curve and bending stress-deflection of bio-board made at other applied pressure is displayed in the appendix.

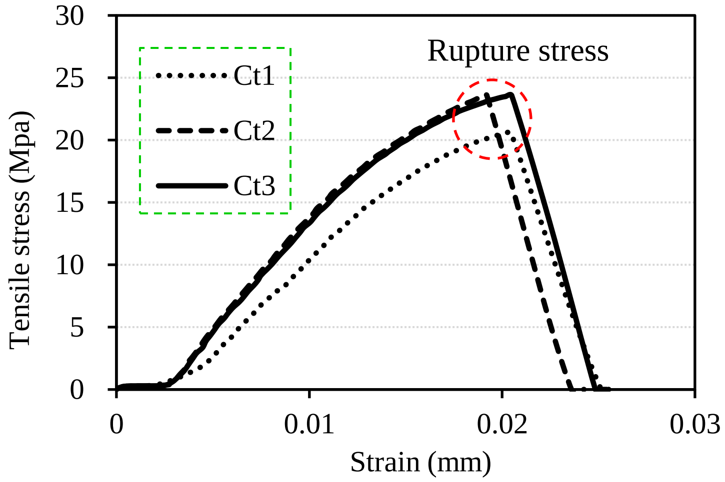


Fig.3-6 Tensile stress-strain curve of bio-board C

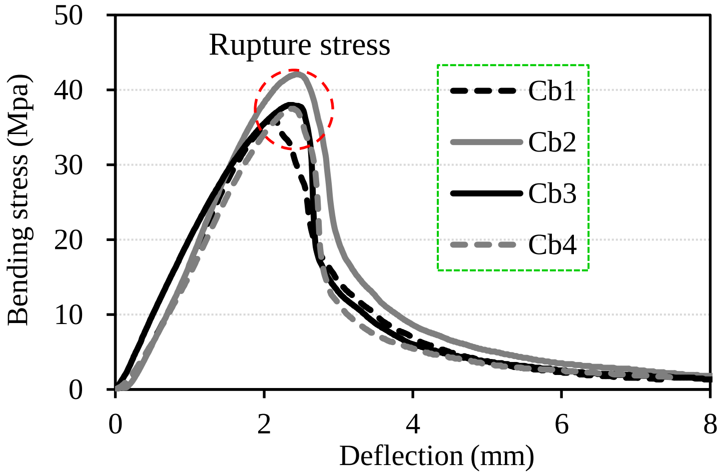


Fig.3-7 Bending stress-deflection curve of bio-board C

Fig. 3-7 exhibits the bending stress-deflection curves of specimens cut from bio-board C, which were coded Cb1, Cb2, Cb3, and Cb4. During the initial stage, it exhibited a similar trend to tensile deformation. When the peak value was reached (between 35MPa and 40MPa), the stress rapidly dropped to a certain value (around 15MPa) and then slowly approached to zero. This was mainly due to some fibers were still connected.

Fig. 3-8 represents the average of tensile rupture stress of bio-boards under different applied pressure. The ANOVA analysis of the data showed statistically significant difference between formulation performance ($p < 0.05$). The tensile rupture stress of the bio-boards was between 15.73 and 22.57MPa, and the maximum tensile rupture stress occurred in bio-board C.

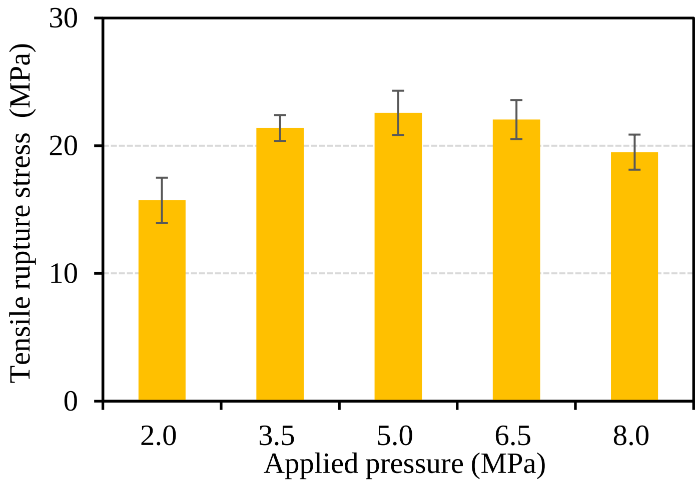


Fig.3-8 Tensile rupture stress of bio-board at different applied pressure

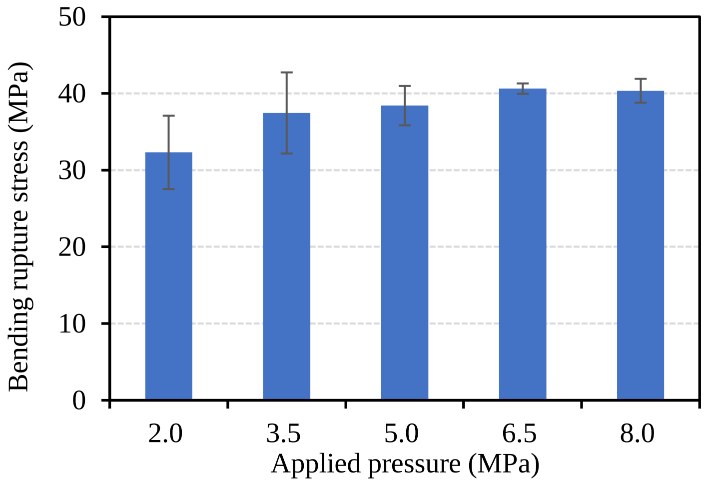


Fig.3-9 Bending rupture stress of bio-board at different applied pressure

Fig. 3-9 represents the average of bending rupture stress of bio-boards under different applied pressure. The ANOVA analysis of the data showed statistically significant difference among formulation performance ($p < 0.05$). With the increase of applied pressure, the bending rupture stress increased from 32.3 to 40.6MPa, the maximum rupture stress occurred in bio-board D, which was made on applied pressure of 6.5 MPa. The static bending properties could not be promoted continuously, especially when the applied pressure great than 6.5 MPa.

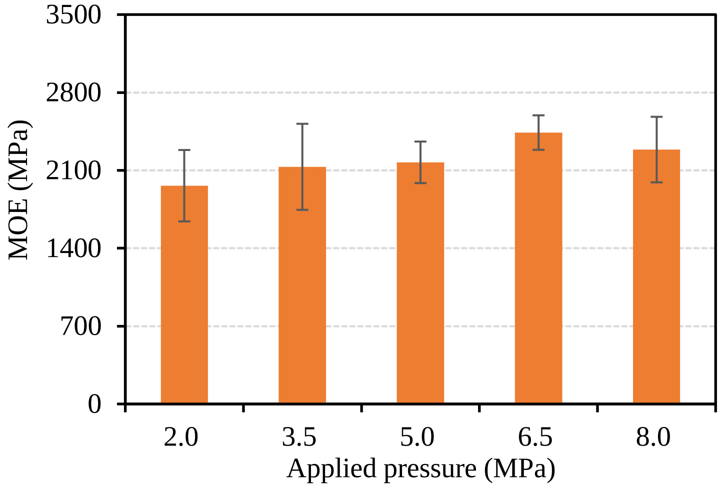


Fig.3-10 MOE of bio-board at different applied pressure

As shown in Fig.3-10, From 2.0 to 6.5 MPa, the MOE of bio-board was first experienced a steady increase from 1961.9 to 2439.6 MPa, and then dropped to 2286.6 MPa at 8.0 MPa. It could be inferred that that the higher applied pressure was not beneficial for the stiffness of bio-board.

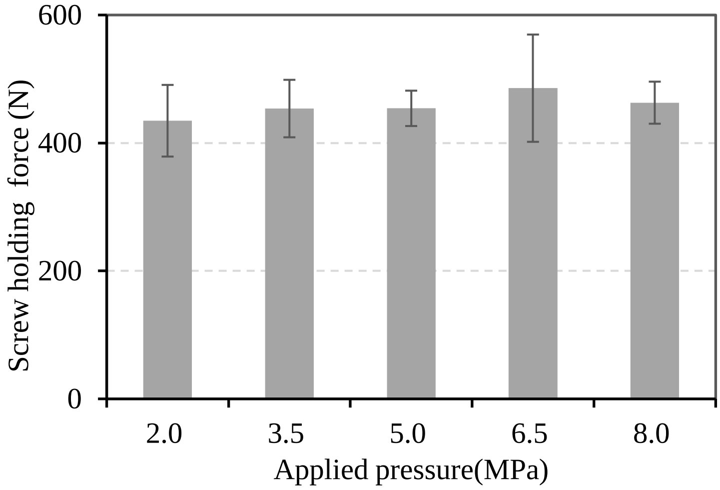


Fig.3-11 Screw holding force of bio-board at different applied pressure.

As shown in Fig. 3-11, the screw holding force of bio-board was in the range of 434.7 to 485.6 N, without pronounced trend along with the rise of applied pressure. An analysis of variance (ANOVA), also found that these values were not significantly different ($p > 0.05$) with the applied pressure.

3.3.3 Dimensional stability performance of bio-board

Water absorption (WA), thickness swell (TS) and linear expansion (LE) of bio-boards at different applied pressure were shown in Fig. 3-12. The WA of bio-boards was range from 87.7% to 97.1%, and the TS was range from 45.8% to 62.0%. The TS increased with the increase of applied pressure. This alteration closely related to the fiber's compactness which affected by the applied pressure. An analysis of variance (ANOVA), found that the results of WA and TS were significantly different (p <0.05), However, when mentioned the LE of bio-board, the results were not significantly different (p >0.05), the LE was basically lower than 2%, and extremely lower than TS.

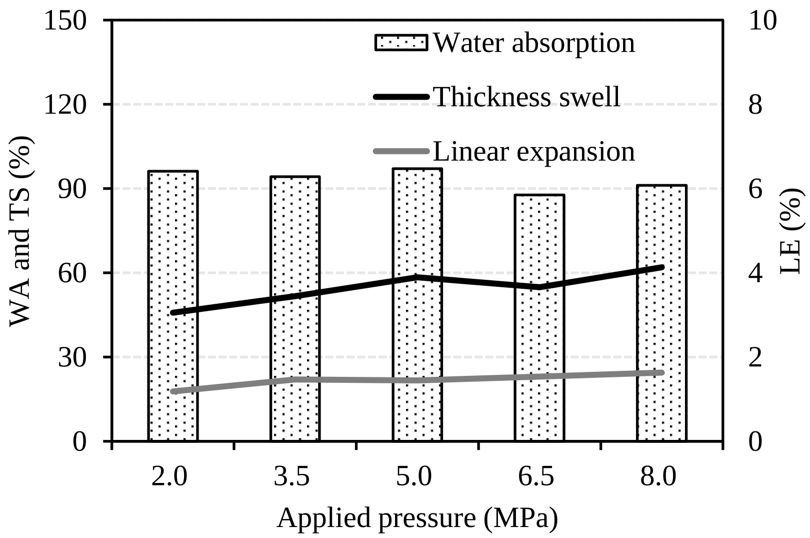


Fig.3-12 WA, TS and LE of bio-board at different applied pressure.

Fig. 3-13 shows the bending rupture stress of bio-boards after water absorption test (in wet state) contrasted with dry state. The wet bending strength was drastically decreased, lower than 3 MPa, and the results were not significantly different (p >0.05) with applied pressure.

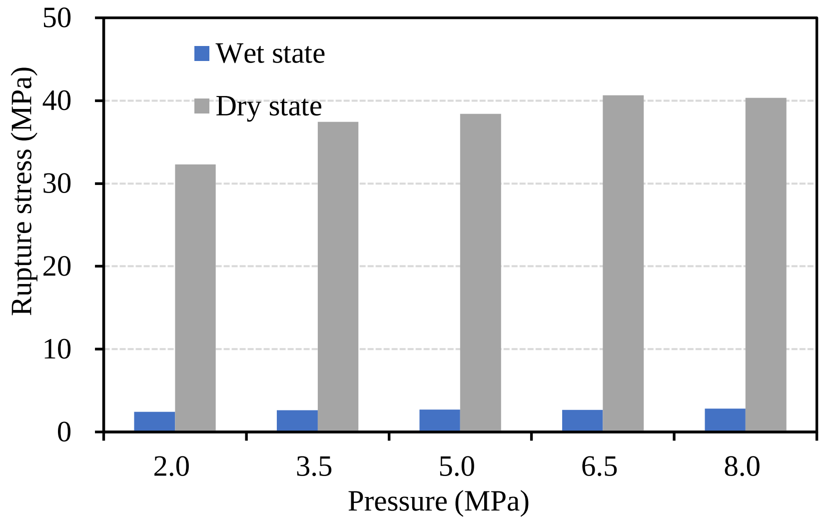


Fig.3-13 Wet bending stress of bio-board at different applied pressure ($p > 0.05$).

3.4 Summary

The bio-boards was made by using soybean straw without synthetic resin, and the effect of applied pressure was investigated, some conclusions was drawn into as follows:

(1) The results indicate that the bio-boards were successfully made and has certain strength. thereby, the making process proposed in this research is valid way to produce board.

(2) The density increased with the increase of applied pressure, whereas, the moisture content shows the opposite tendency. Mechanical properties of bio-board, such as bending properties, tensile properties, and screw holding force, were closely related to the density and moisture content affected by the applied pressure.

(3) The TS value of bio-board increased with the increase of applied pressure, and the WA of them was over 85%, overall, bio-board has poor water-proof properties, and the bending performance is also significantly decreased after absorbing water.

Chapter 4. Effect of forming time on properties of bio-board manufactured by soybean straw and corn straw

4.1 Introduction

The main chemical compositions of soybean straw and corn straw are shown in Table 1 [41][42]. The cellulose of corn straw was slightly higher than that of soybean straw. However, the lignin in corn straw was 58.55% of that in soybean straw, but the hemicellulose in corn straw was 2.2 times of that in soybean straw. These differences might significantly influence the properties of produced bio-board. Therefore, the two kinds of crop straw were chosen to produce bio-board, and it's mechanical and physical properties were compared to investigate the influence of chemical compositions. The research about the effect of forming time on properties of fiberboard was reported by previous literature[43][17]. However, it's mainly conducted in the dry-forming process of fiberboard. The forming time was an essential experimental parameter for the wet-forming process as well. Thus, forming time was selected as a parameter to evaluate the properties of bio-board produced in wet-forming process.

Table 6 Main chemical compositions of soybean straw and corn straw.

Ingredient	Cellulose	Hemicellulose	Lignin
Soybean straw	39.93%	14.15%	23.31%
Corn straw	42.25%	31.78%	13.65%

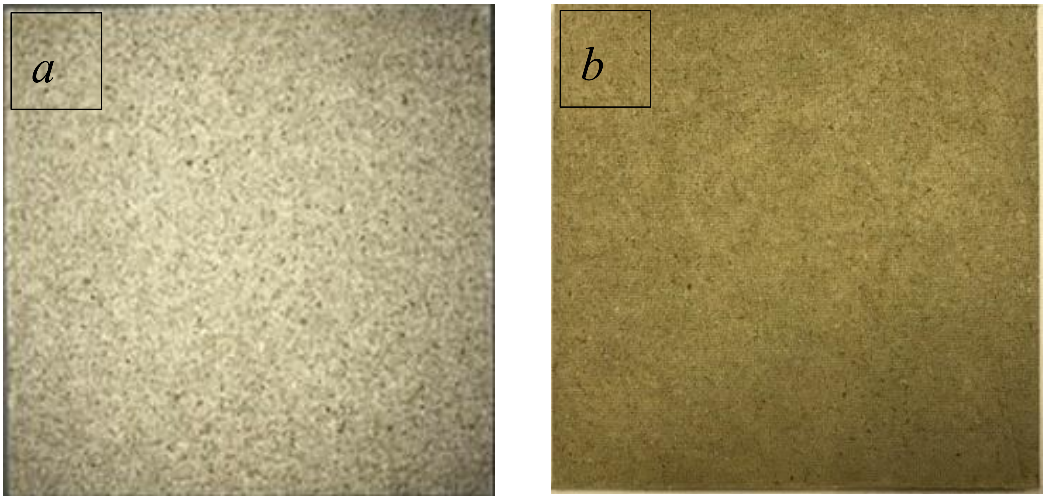
4.2 Materials and method

Bio-board was made under various conditions shown in Table 2. The condition of heating temperature was 140 °C, and applied pressure was 5.0 MPa. The forming time was 0.5 to 2.5 h with an interval of 0.5 h.

Table 7 Making condition of bio-board.

Items	Heating Temperature (℃)	Applied pressure (MPa)	Forming time (h)
Corn straw	140	5.0	0.5-2.5
Soybean straw	140	5.0	0.5-2.5

Finally, two pieces of bio-boards were produced under each forming condition. Fig. 1 shows the corn straw bio-board and soybean straw bio-board samples. The color difference between the two kinds of bio-board is associated with the chemical compositions inside raw materials. After that, the mechanical tests and water absorption tests were carried out to investigate the properties of the bio-boards.



a. Soybean straw bio-board. b. Corn straw bio-board

Fig.4-1 Bio-board produced by corn straw and soybean straw.

4.3 Results and discussion

4.3.1 Density and moisture content of bio-board

Fig. 4-2 and Fig. 4-3 show the values of density and moisture content of the bio-boards manufactured from corn straw and soybean strew, respectively. Both time and straw type are not statistically significant in density of bio-boards, whereas they are statistically significant in moisture content of bio-boards. The density of these two kinds of bio-boards were almost similar and distributed in a narrow range of 1.05 to 1.10 g/cm^3 under different forming time.

The density was mainly affected by the applied pressure rather than forming time.

The bio-board produced in 0.5 h had the highest moisture content values of 6.0% for soybean straw bio-board and 5.0% for corn straw bio-board. Further, the moisture content decreased with the increase of forming time firstly, then almost kept in constant of 4.1% for soy and 2.9% for corn.

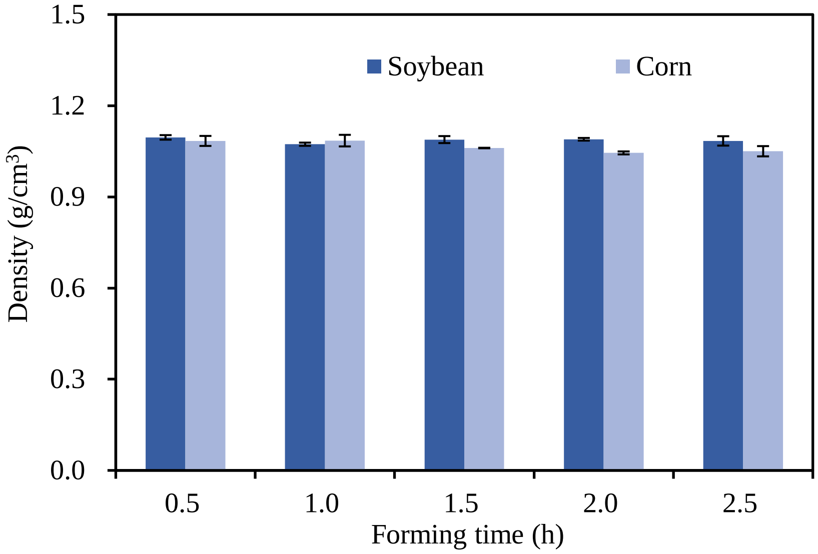


Fig.4-2 Density of bio-board made at different forming time.

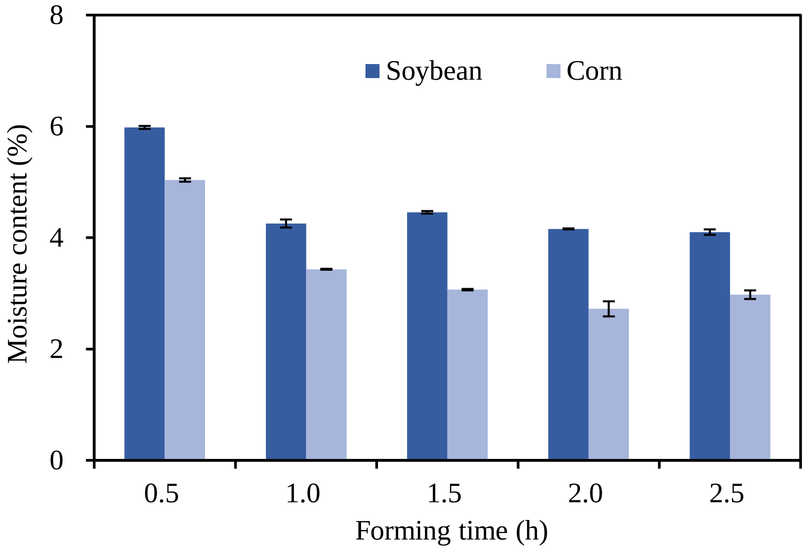


Fig.4-3 Moisture content of bio-board made at different forming time. ($p < 0.05$)

4.3.2 Mechanical properties of bio-board

The mechanical performance of bio-board was usually expressed by the bending rupture stress and tensile rupture stress. The average value of bending rupture stress was plotted in Fig.4-4 The bending rupture stress of soybean straw bio-board was slightly increased from 39.3 to 43.2 MPa when the forming time raised from 0.5 to 2.5 h. The bending rupture stress of corn straw bio-board was between 31.7 and 46.0 MPa, which increased firstly, then had a small drop for the board made using 2.5 h. Except for the bio-board made at 0.5 h which has a lower initial value, and the fluctuation was higher than that of soybean straw bio-board.

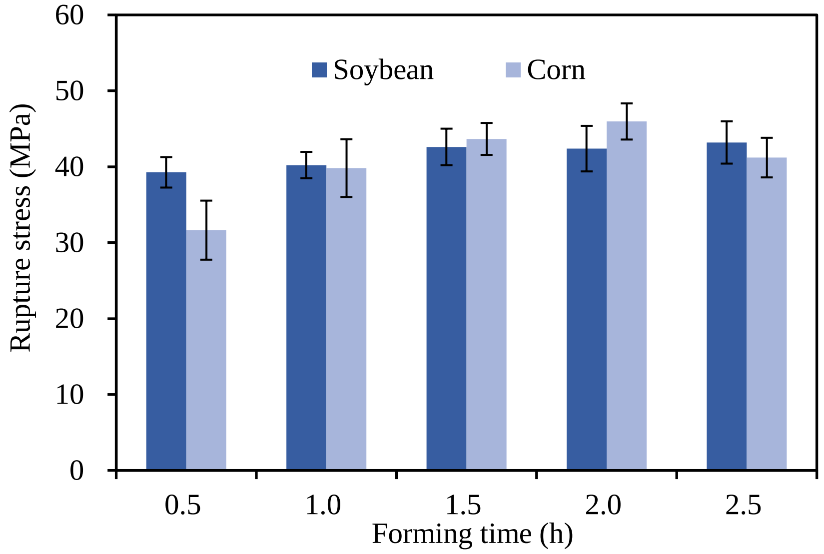


Fig.4-4 Bending rupture stress of bio-board made at different forming time

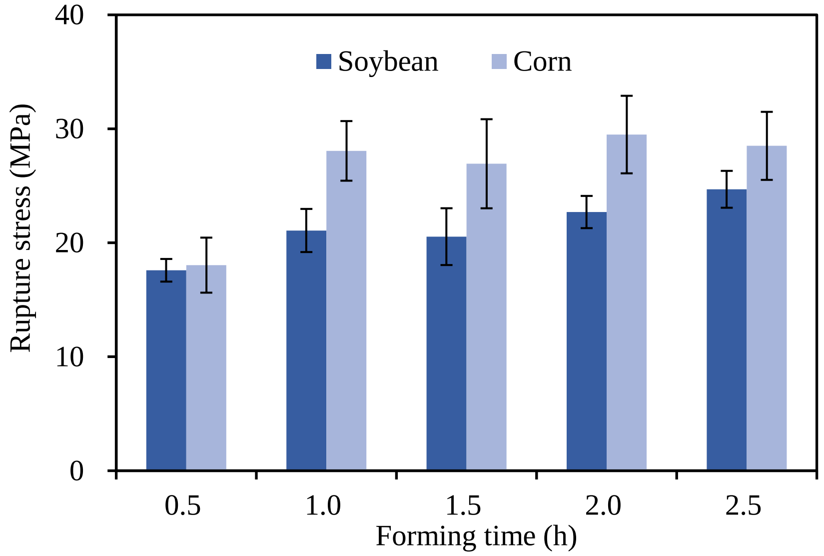


Fig.4-5 Tensile rupture stress of bio-board made at different forming time.

Fig.4-5 shows the tensile rupture stress of two kinds of bio-boards. The tensile rupture of the soybean straw bio-board increased slightly with the increase of forming time. The tensile rupture stress of soybean straw bio-board varied from 17.6 to 24.7 MPa. The tensile rupture stress of corn straw bio-board varied from 18.0 to 29.5 MPa. Except for the corn straw bio-board made at 0.5 h, tensile rupture stress shows the same level for other forming time condition, and it was higher than that of soybean straw bio-board.

The soybean straw contains much more lignin, and the high content of lignin was beneficial for the mechanical properties according to Bouajila et al., [44], and Anglès et al., [33]. Furthermore, Lignin is relatively stable compared with hemicellulose at 140 ℃. Therefore, soybean straw bio-board has a higher initial value, and the overall change was less than the corn straw bio-board. The corn straw bio-board was closely related to the polysaccharide of hemicellulose. Polysaccharide cross-linking reactions were in favor of fiber bonding ability. This mechanism could improve the mechanical properties with the prolonged forming time. However, the mechanical properties could not be promoted continuously, especially when the forming time was greater than 2 h.

4.3.3 Dimensional stability performance of bio-board

WA, TS and LE of two kinds of bio-boards were shown in Fig.4-6 and Fig.4-7. The WA of soybean bio-boards was ranging from 97.2% to 123.4%, and the TS was ranging from 66.2% to 97.8%. Both WA and TS decreased firstly, then almost kept in constant with the increase of forming time for soybean straw bio-board. The corn straw bio-board made using 0.5 h had the highest WA of 101.5%, which was about 35% higher than other forming time. However, the value of TS was between 60.3% and 65.4% with no remarkable difference for each forming time. Overall, the WA and TS of corn straw bio-board lower than those of soybean straw bio-board for each forming time condition. Straw' type was statistically significant for WA and TS of bio-boards. Owing to the corn straw contains more cellulose, which was difficult to dissolve in water. On the other hand, the hemicellulose consists of xylose, arabinose and other polysaccharides which acted as a binder at 140 ℃. Although, the bio-board made by corn straw fiber has better performance to resist the permeability of water than that of soybean straw bio-board. The LE of two kinds of bio-boards was lower than 2% with no statistically difference among different forming time and different materials, and extremely lower than TS. The huge difference between LE and TS was caused by the fact that the bio-board in the direction perpendicular to the side surface was pressed during the forming stage.

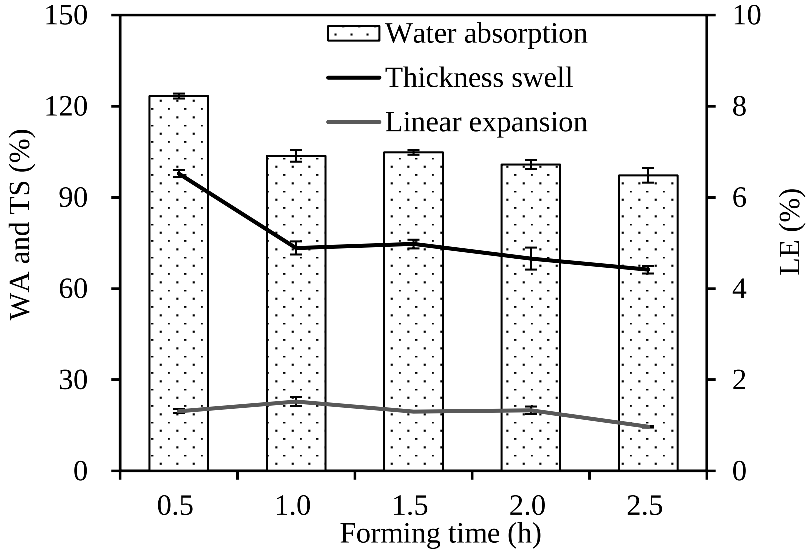


Fig.4-6 WA, TS and LE of bio-board at different forming time. (soybean straw)

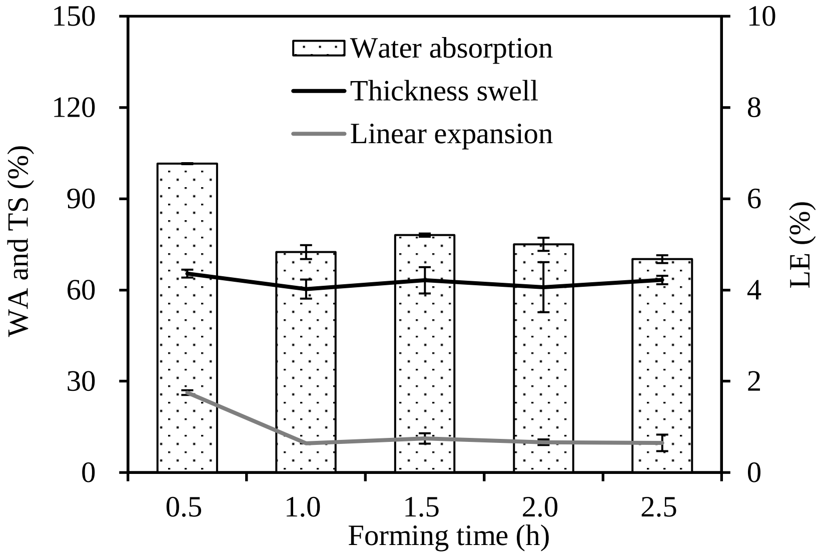


Fig.4-7 WA, TS and LE of bio-board at different forming time. (corn straw)

4.4 Summary

In this chapter, the bio-board was produced by using soybean straw and corn straw without adding any binder at different forming time. Several property experiments were carried out to investigate the influence of forming time and the chemical compositions of straw on bio-board. The conclusions are as follows:

(1) Except for the bio-board made at 0.5 h that has relatively low strength and water-proof performance, the performance at other forming time condition did not have significant difference both soybean straw and corn straw bio-board.

(2) The bio-board made of corn straw had better performance in resisting the permeability of water than soybean straw bio-board. The fluctuation of bending rupture stress for corn straw bio-boards was more significant than that for soybean straw bio-board, and the tensile rupture stress for corn straw bio-board were mostly higher than that for soybean straw bio-board. The properties of the two kinds of bio-boards were closely related to the chemical compositions of raw materials.

Chapter 5. Effect of heating temperature on properties of bio-board

5.1 Introduction

The self-bonding on non-resin fiberboard is mainly accomplished using naturally occurring materials. According to Bouajila et al.[44], the bonding strength of binderless boards may be due to lignin-lignin and lignin-polysaccharide cross-linking reactions that occur at high temperature and deformation of the system under pressure. Winandy et al. [45]evaluated changes in carbohydrate composition and structure as a function of multiple press temperatures. For most mechanical properties, it appears that very little degrade occurs until mat temperatures exceed 150 °C. Salvadò et al. [46] tried to maximize the properties of binderless boards using steam-exploded Miscanthus sinensis by pressing them at temperatures ranging from 195 to 245 ℃ and have obtained satisfactory results. The generation of simple sugars from the degradation of hemicelluloses at 170 ℃ and the partial degradation of cellulose around 220 ℃ to produce simple sugars have been reported to contribute to bonding in binderless boards made from steam-exploded materials [47]. Heating temperature seems to be the most influential parameter of lignin changes, subsequently affecting the properties of binderless board. A similar conclusion has been reported by Hashim et al.[48], Gul et al.[49], and Wang et al[50].

5.2 Materials and method

In this part 15 pieces of bio-boards in five categories were made in several conditions. There was increase of temperature from 110 to 230 ℃ at intervals of 30 ℃. The condition of pressure was 5 MPa, which was accepted to be the optimum in some preliminary tests. The time of pressing and heating was 30 min in the process of forming. The produced bio-boards are shown in Fig.5-1. They exhibited smooth surfaces and colors ranging from light brown to dark brown.

Table 8 The making condition of bio-board.

Item.	Heating Temperature (℃)	Applied pressure (MPa)	Forming time (min)
A	110	5.0	30
B	140	5.0	30
C	170	5.0	30
D	200	5.0	30
E	230	5.0	30

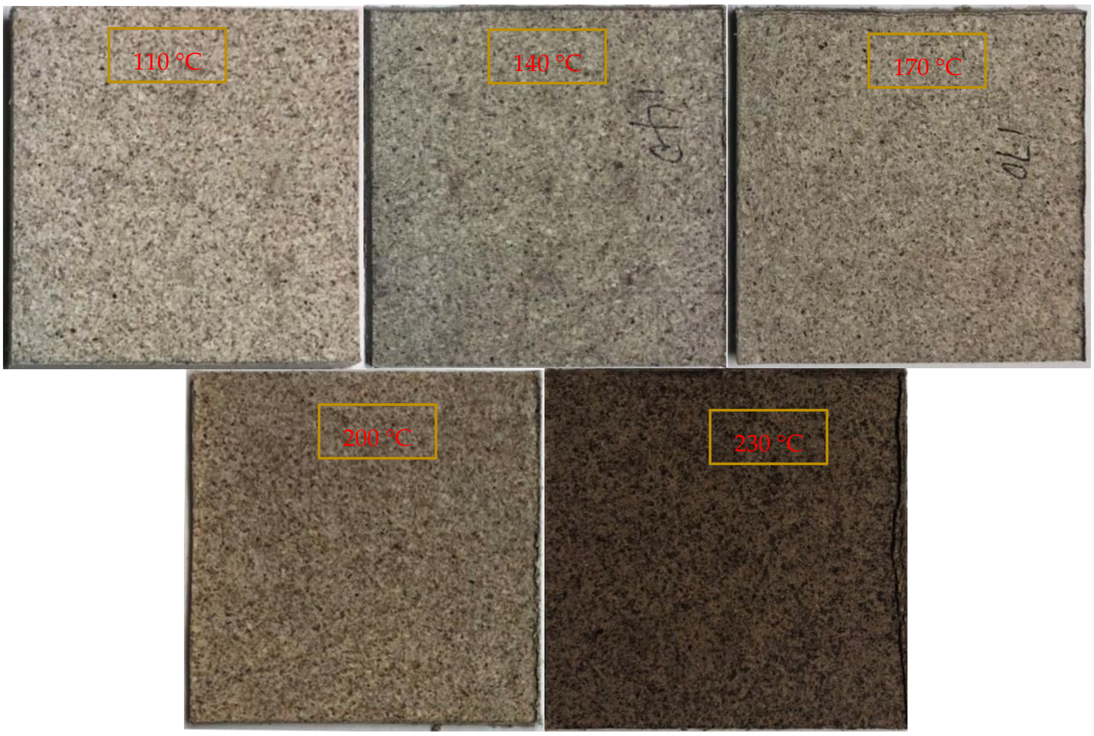


Fig.5-1 Bio-board produced at different heating temperature.

5.3 Results and discussion

5.3.1 Thermal properties

TGA results of bio-board are shown in Fig 5-2, which represents weight loss curves (TG) and derivative thermogravimetric (DTG) curves. The TG curves show an initial decrease in weight while the temperature is lower than 110 °C. This weight loss could be caused by moisture in the soybean straw powder diffused in the air by heat. After that, the weight does not change substantially between 110 and 200 °C. From 200 to 400 °C, the weight decreases from 93.1% to 22.2%, and derivate weight loss rate achieved the maximum value at a temperature of 382 °C. Macedo et al. [51] reported the sequence of thermal degradation of the plant biomacromolecules was hemicellulose (200-260 °C), cellulose (240-350 °C), and lignin (280-500 °C). The thermal decomposition of hemicellulose was expected to occur in the process of production of bio-board at 200 and 230 °C. When the temperature is above 400 °C, the rest of the lignin tends to gradually degrade until about 14% of the ash was left.

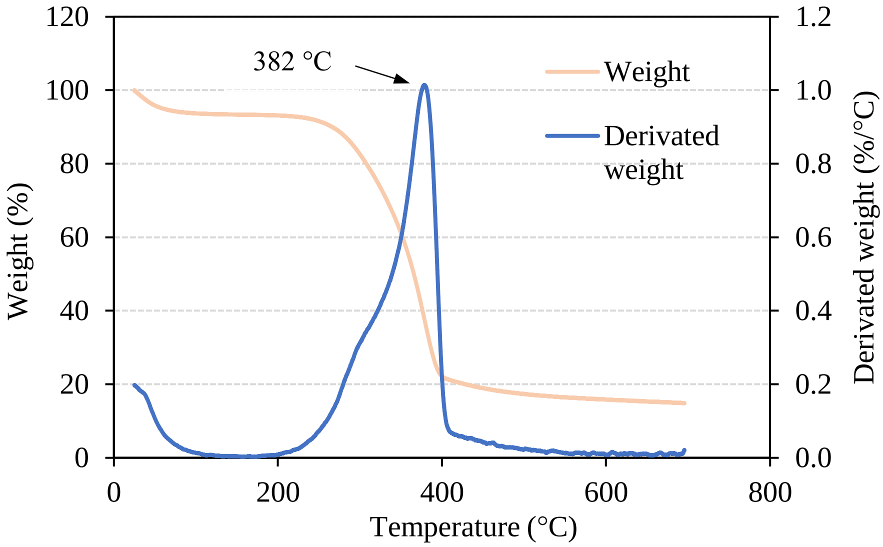


Fig.5-2 TG and DTG curves of the samples.

5. 3.2. Functional group analysis

The FTIR spectra of the produced bio-board at different heating temperatures are shown in Fig.5-3. The samples of bio-board do not have significant difference in accordance with the peak position of the spectrum. The absorption band around the 3400 cm^{-1} indicates –OH stretching. It indicates the existence of hydrogen bonds or –OH in aliphatic aromatic compounds of the samples[52]. The peak around the 2900 cm^{-1} is related to the C–H stretching vibration. Two peaks are generated between 1680 cm^{-1} and 1750 cm^{-1} which might be caused by the conjugating of the benzene ring with the hydroxyl (–OH) or amino group ($–NH_2$), resulting in a ring-absorbing peak[53]. A series of peaks are generated between 1000 cm^{-1} and 1500 cm^{-1} which is associated with the stretching vibration of C–O, C–C, and the bending vibration of C–OH. It is worth noting that the absorbance at 3400 cm^{-1} at the temperature of 200 and 230 °C is higher than that at other temperatures. The results could be attributed to the degradation of hemicellulose as reported by Bledzki et al. [55]. Because hemicellulose was a hydrophilic substance, the reduction of it could be potential evidence to the highly waterproof performance of the bio-board made at high temperature.

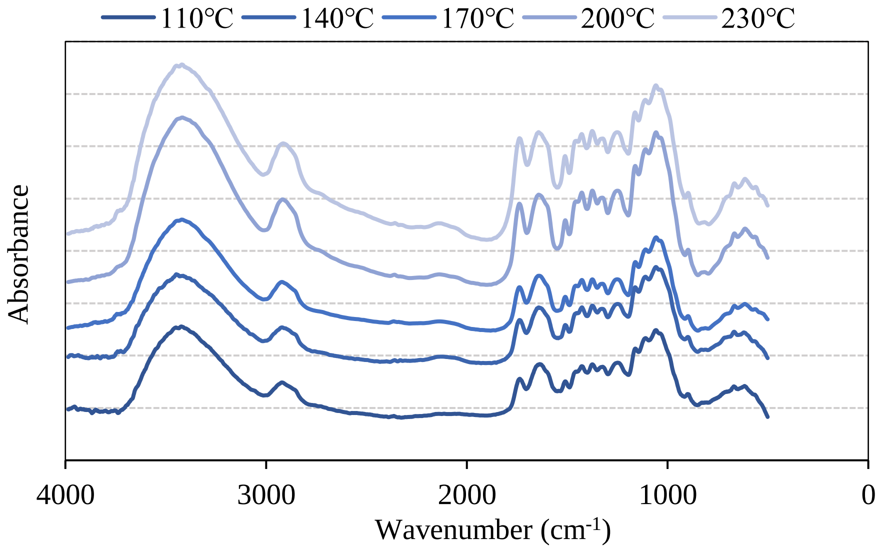
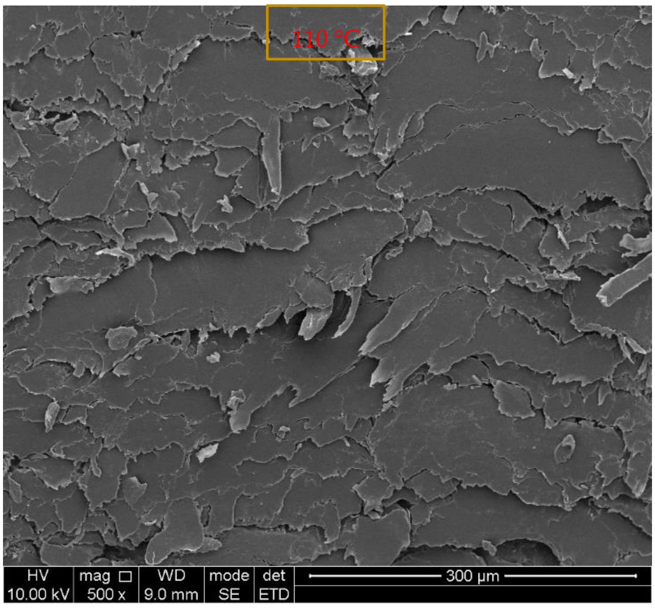


Fig.5-3 FTIR spectra of the samples at different heating temperature.

5.3.3. SEM analysis

The section morphology of the bio-board manufactured from 110 to 230 °C is shown in Fig.5-4. The section of the bio-board made at 110 °C showed continuous narrow irregular fissure existing among the observed zone clearly, and some scaly shape covers the surface. Moreover, uneven delamination means a heterogeneous fiber distribution inside the bio-board, and this might bring about poor mechanical performance externally. With the increase of heating temperature, the cross-section texture becomes dense and uniform. When the temperature reaches 200 °C, it exhibits a smoother cross-section texture, which implies a superior mechanical property. However, at 230 °C, there exist holes and signs of thermal degradation in the cross-section, indicating that the excessively high temperature is less promoted for the improvement of the mechanical properties. Notably, the last two pictures in Fig.5-4 magnify the bio-board samples made at 200 and 230 °C to 10000 times. It had been observed that the surface of fiber was smooth at 200 °C, but the surface was rough and full of fine granules after pyrolysis at 230°C.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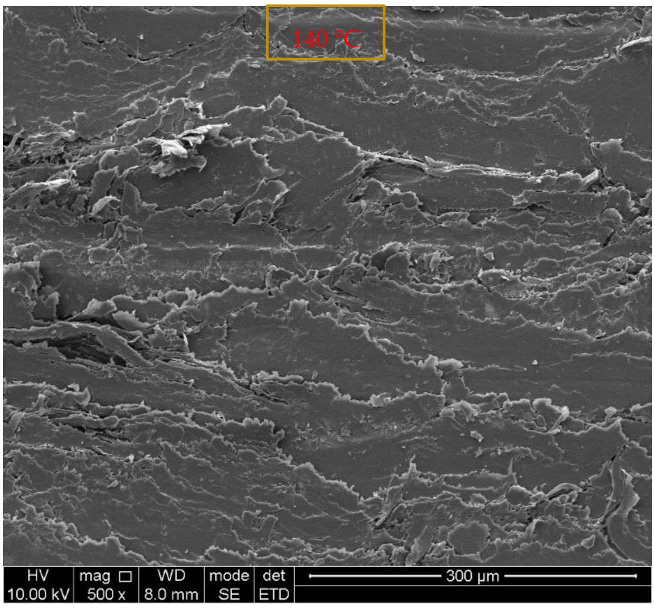
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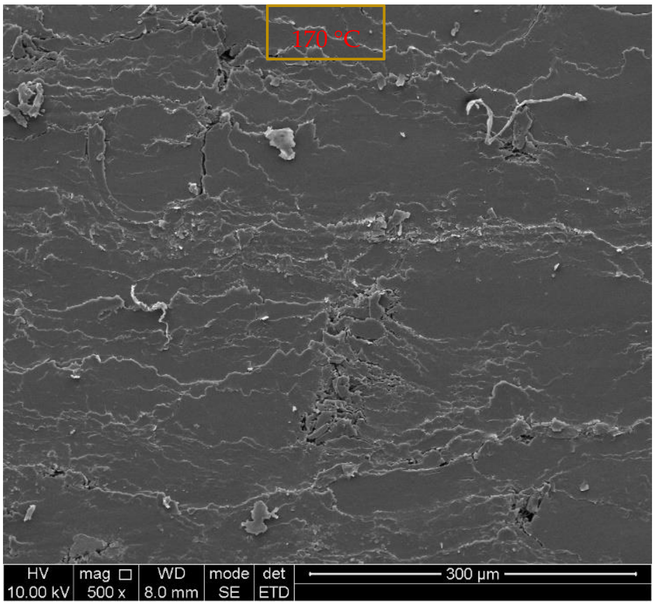
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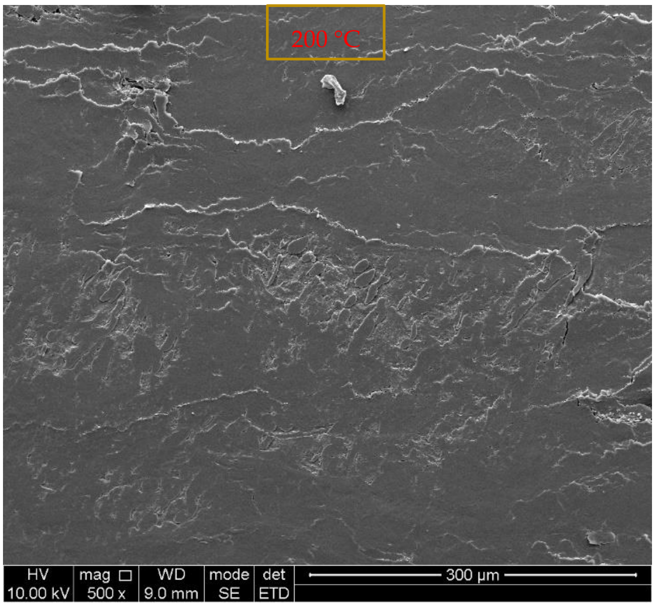
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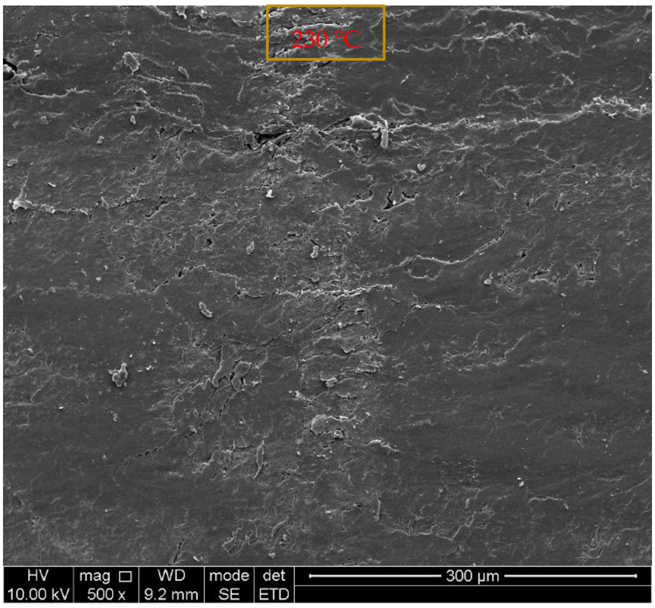
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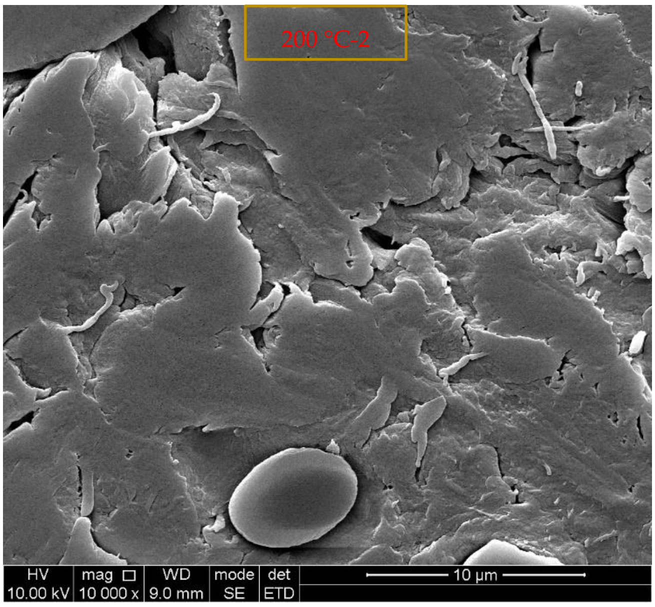
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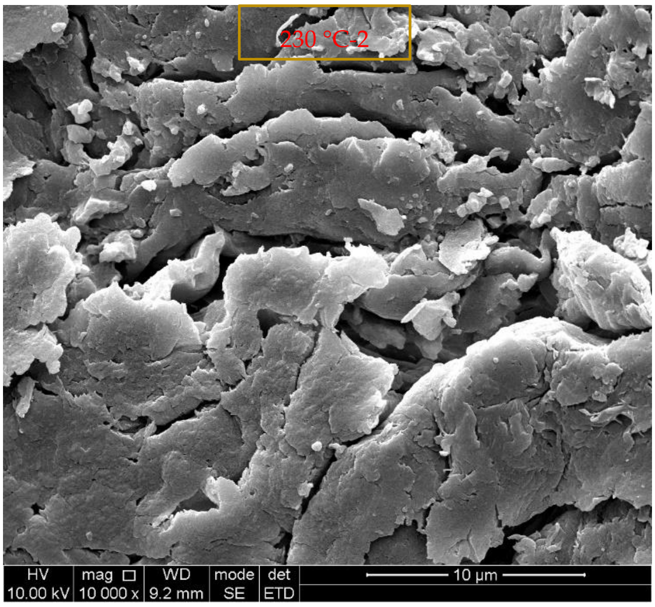


Fig.5-4 Section morphology of bio-board at different heating temperature

5.3.4. The physical properties of bio-board.

Fig.5-5 shows the density of the bio-board produced at different heating temperature. The density at different heating temperature is almost the same as 1.1 g/cm^3, except for the density 0.88 g/cm^3 at 110 °C. Fig.5-6 exhibits the moisture content of bio-board at each heating temperature. It is obvious that the moisture content decreases with the rise of temperature. When the temperature is 110 °C, the moisture content is 12.5% larger than that of others. It could be inferred that there are many tiny voids filled with air and free water molecules inside the bio-board made at 110 °C. The SEM results also show that there exists some fissure in the samples. Therefore, the density of bio-board made at 110 °C is lower than the others.

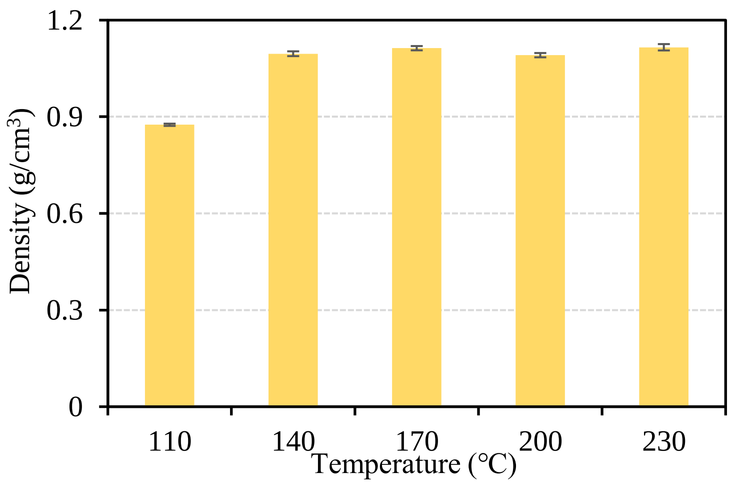


Fig.5-5 Density of bio-board at different temperature.

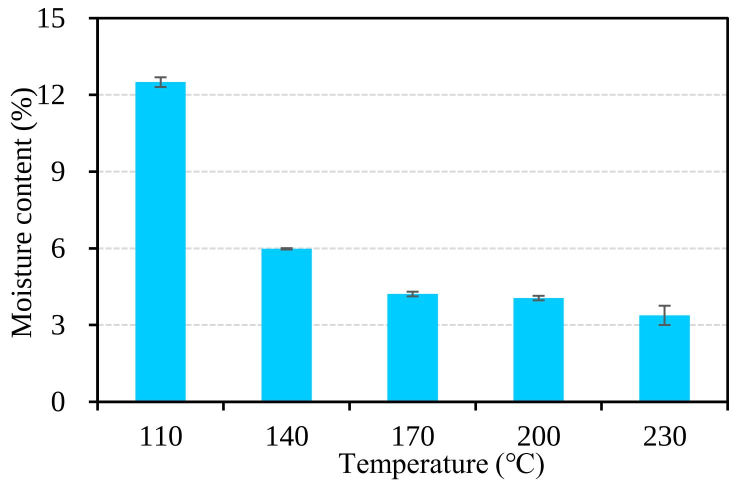


Fig.5-6 Moisture content of bio-board at different temperature.

5.3.5. The mechanical properties of bio-board

To investigate the strength of bio-board produced in this study, a bending test and tensile test were carried out. Fig.5-7 and 5-8 represent the tensile rupture stress and the bending rupture stress of bio-board, respectively. As shown in Fig.5-7, the tensile rupture stress is between 8.4 and 24.4 MPa, increasing with the rise of heating temperature. As shown in Fig.5-8, the bending rupture stress of bio-board produced at 110 °C is 15.5 MPa which was significantly lower than the others. From 140 to 230 °C, the bending rupture stress first experienced a slow increase from 39.3 to 42.1 MPa and dropped to 35.8 MPa at 230 °C. The JIS A5908 hardboard S35 type required minimum bending rupture stress of 35 MPa. Nearly all the bio-board met the requirement for bending rupture stress, except the bio-board made at 110 °C.

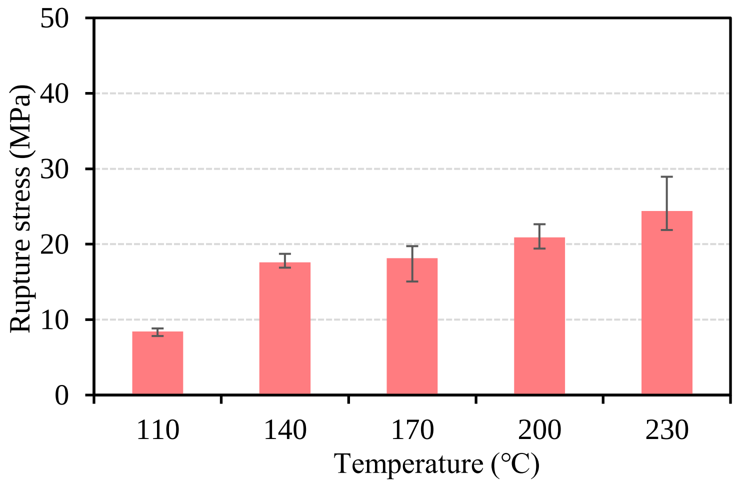


Fig.5-7 Tensile rupture stress of bio-board at different temperature.

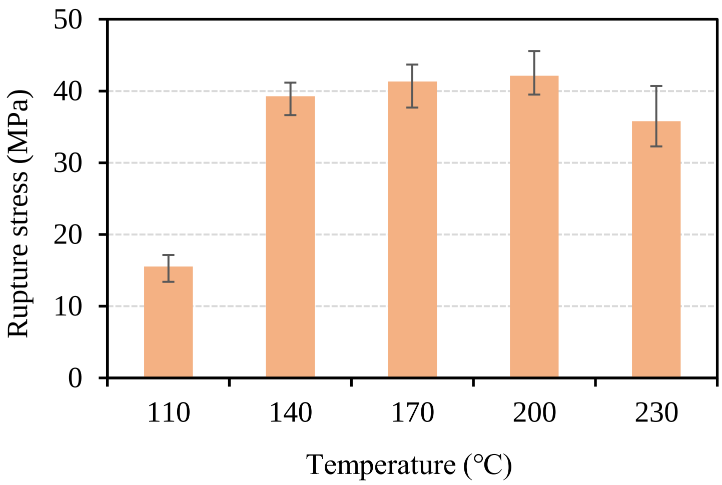


Fig.5-8 Bending rupture stress of bio-board at different temperature.

According to the moisture content discussed above, it is clear that the higher water content was the reason that both bending and tensile strength of the bio-board pressed at 110 °C had lower rupture stress. From 140 to 200 °C, the moisture content of bio-board decreased as shown in Figure 8, and the reduction of free water molecules represented the direct connection among fiber macromolecules through hydrogen bonding, or van der Waals forces were enhanced. Moreover, a softening of lignin during the forming process also occurred, and it was beneficial for bonding the fiber together more tightly. The lignin glass transition temperature was directly related to the moisture content according to the previous literature [56][57]. The wet-forming method was used in this study. In the process of forming, the high moisture content of the bio-board mat would significantly reduce the glass transition temperature of lignin, and the softened lignin acted as a binder. Therefore, the bending strength and tensile strength slightly increased. Bio-board produced at 230 °C had a minimum moisture content of 3.4%, as shown in Figure 8. However, it is worth noting that the average tensile rupture stress increased by 16.5% compared with that of 200 °C, but the average bending rupture stress decreased by 15.1%. The results might be related to the chemical changes of material. According to Macedo et al.[51], the hemicellulose would be pyrolyzed above 200 °C, and the pyrolysis products underwent condensation or polymerization with the temperature rise. At 230 °C, the condensation reaction and plasticization brought about an embrittling and hardening of the bio-board, which was not favorable to the bending strength of the bio-board.

The fiberboard used in furniture and construction often requires screw fastening installation, so it is significant to evaluate the screw-holding force of bio-board. Fig.5-9 represents the screw-holding force of bio-board. The screw-holding forces were between

224.9 and 390.4 N. The screw-holding force of bio-boards were greater than 350 N, except for that made at heating temperature of 110 °C. Generally, the screw-holding forces were similar to the bending strength as described above.

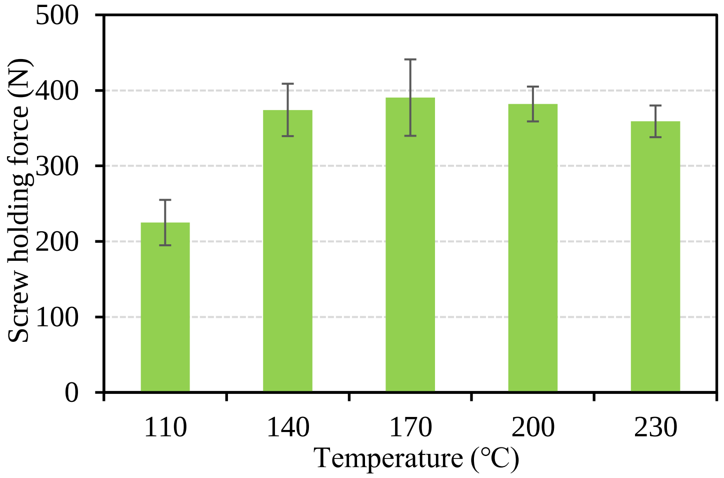


Fig.5-9 Screw holding force of bio-board at different temperature.

5.3.6 Dimensional stability performance of bio-board

The results of WA and TS are shown in Fig.5-10. From 110 to 140 °C, the WA increased from 105.4% to 123.4% first, and then decreased from 123.4% to 41.5% after 140 °C. Similarly, the TS increased from 46.1% to 97.8%, then decreased from 97.8% to 23.5%. The dimensional stability performance of bio-board increased from 140 to 230 °C. As described in JIS A5908, the WA of hardboard S35 type should not be greater than 35%, and none of the bio-board could met the JIS standard for this parameter.

Both WA and TS increased during the heating temperature changing from 110 to 140 °C. This phenomenon might be caused by the following two reasons. First, the interspace among fibers of bio-board pressed at 110 °C had higher moisture content as shown in Figure 8; therefore, it was difficult to absorb more water than that of the bio-board pressed at 140 °C. Secondly, the bio-board was pressed at 140 °C with a higher density than that of 110 °C, and fibers huddle together tightly. After the WA test, the hydrogen bonds among the fibers opened, and the fiber swelled and the voids among fibers expanded significantly. Therefore, the bio-board pressed at 140 °C could absorb more water than that at 110 °C, and the TS rate was changed more obviously.

From 140 to 230 °C, WA and TS gradually decreased synchronously. The phenomenon could be attributed to the occurrence of the following chemical reactions. The first reason was the pyrolysis of hemicellulose. Owing to poor thermal stability of hemicellulose, the pyrolysis would first appear with the increase of heating temperature. Moreover, hemicellulose was a hydrophilic substance, and its reduction would improve the waterproof properties to some extent. The second reason was the condensation reaction of lignin. When the heating temperature was above 200 °C, the condensation reaction of lignin occurs, and the substance produced by the condensation reaction was black and hardly soluble in water, so the moisture content was greatly reduced after the water-soaking test.

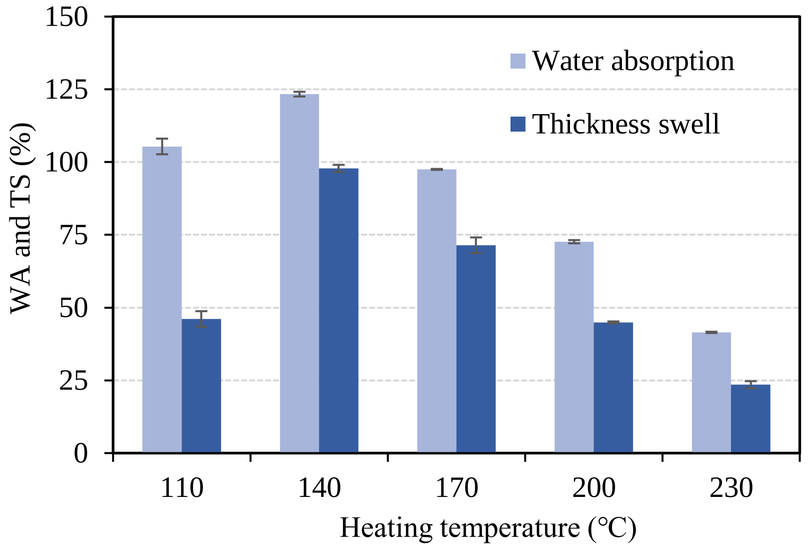


Fig.5-10 The WA and TS of bio-board at different temperatures.

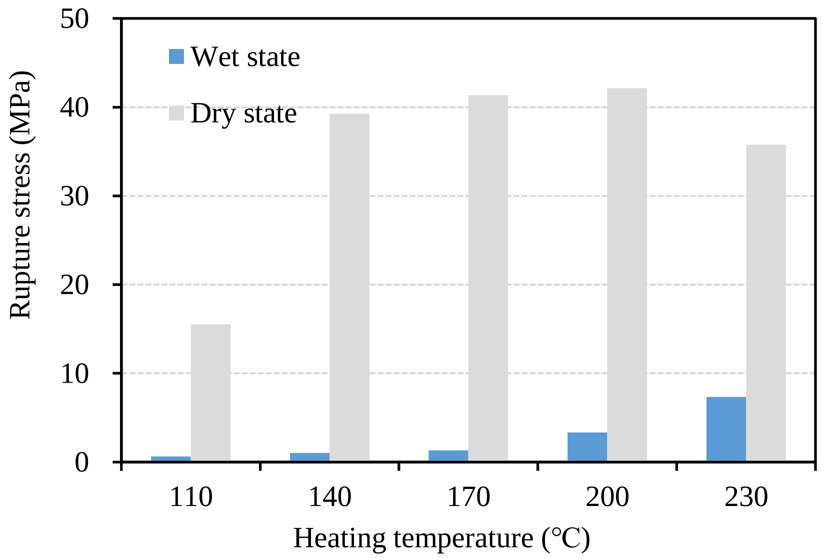


Fig.5-11 Wet bending stress of bio-board at different heating temperature.

Fig. 5-11 shows the bending rupture stress of bio-boards after water absorption test (in wet state) contrasted with dry state. The wet bending strength was increased from 0.64 to

7.34 MPa, and the results were significantly different ($p > 0.05$) with heating temperature. The bio-board made at high temperature has better performance to resist the permeability of water, therefore, the wet bending properties of specimens after water absorption test was improved at high heating temperature.

5.4 Summary

In this chapter, bio-boards were made by using soybean straw without any petroleum-based resins. Moreover, the influence of heating temperature on mechanical properties and dimensional stability performance of bio-board was investigated. Some conclusions were revealed as follows:

In general, with the increase in temperature, there was a decrease in moisture content, the softening of lignin, and the pyrolysis of hemicellulose, which is beneficial to the improvement of the mechanical properties of the bio-board. However, the excessive heating temperature, especially at 230 °C, does not significantly promote improvement to most mechanical properties. On the other hand, the dimensional stability of the bio-board is greatly improved from 140 to 230 °C.

Chapter 6. Effect of fiber length on properties of bio-board

6.1 Introduction

The properties of fiberboard could be significantly affected by fiber geometry, including fiber size and fiber shape. Miyamoto et al. [58] revealed that with the decrease of particle size the linear expansion increased under different density level. It was considered that the out-of-plane orientation angle of the particles affected the LE of the boards. Rokiah Hashim et al. [59] made binderless particleboard using fine particles and strands of oil palm trunks, respectively. The results showed that board made by strands had better performance both mechanical and dimensional stability. It was concluded that larger contact area of longer and thinner strands or a particle showed higher properties. However, according to the research of Osarenmwinda and Nwachukwu, it was claimed that with the decreased of particle size, the value of MOE, MOR, and IB was decreased, while the value of WA and TS was increased [60]. Similar results were revealed by Ali et al[61]. Short fibers packed more compactly preventing water from penetrating into matrix, which was beneficial for improve the dimensional stability of board. However, the long fiber mingled with each other, and the interweaving between it could improve the friction between fibers, Therefore the mechanical properties of long fiber board performed better than short fiber board.

As mentioned above, the mechanism of the fiber geometry on board properties was not clearly figured out. The objective in this chapter is to investigate the effect of fiber length on no-resin bio-board. To evaluate the influence of fiber length on properties of bio-board. Soybean straw fiber was prepared with the three categories of fiber lengths: long fiber (longer than 2mm), short fiber (shorter than 1mm) and mixed fiber (mix of long fiber and short fiber). Further, applied pressure in the forming process was an influential factor in the properties of bio-board. Bio-board manufactured with three fiber length categories and different pressure was applied to understand the effects of these factors on mechanical and dimensional properties of bio-board.

6.2 Materials and method

Three different sizes of fiber pulp were generated as shown in Fig. 1(a, b, and c). It's worth noting that the short fiber in Fig.1a could pass through 2 mm sieves, and the refining time of it for approximately 30 minutes. The mixed fiber in Fig.1c was the products generated at the refining time of 20 minutes. The long fiber in Fig.1b with a length of more than 2 mm, which were the product after removing the tiny fibers from the mixed fibers through flowing water by 2 mm sieves.

(a) short fiber; (b) long fiber; (c) mixed fiber.

Fig.6-1 Three different size of soybean fiber.

Table 9 The making condition of bio-board.

No.	Refining time (min)	Fiber length	Heating temperature (℃)	Applied pressure (MPa)	Forming time (h)
A	30	< 1mm	110	2.0, 3.5, 5.0, 6.5, 8.0	2
B	20	>2mm	110	2.0, 3.5, 5.0, 6.5, 8.0	2
C	20	mixture	110	2.0, 3.5, 5.0, 6.5, 8.0	2

In this chapter bio-boards were made under the conditions of table 10. There was increase of applied pressure from 2 to 10 MPa. The condition of heating temperature was 110 ℃, and the forming time was 2 h. The produced bio-boards are shown in Fig. 6-2. The surface of long-fiber bio-board and mixed-fiber bio-board is rough, and the separated fibers can be observed on the surface.

a. short fiber. b. long fiber. c. mixed fiber.

Fig.6-2 The bio-board produced at different fiber length.

6.3 Results and discussion

The trends of change in density are shown in Fig.6-3. The density tends to increase for all the three types of fiber length when the applied pressure increased from 2MPa to 10MPa. The density of bio-boards made from long fibers is similar to that of the short fibers, whereas the density of bio-boards made from mixed fibers is higher than the others mentioned above in most cases. It could be inferred that the short fibers are stuffed among the long fibers, which were conducive to from a dense structure inside of the bio-board made from mixed fibers.

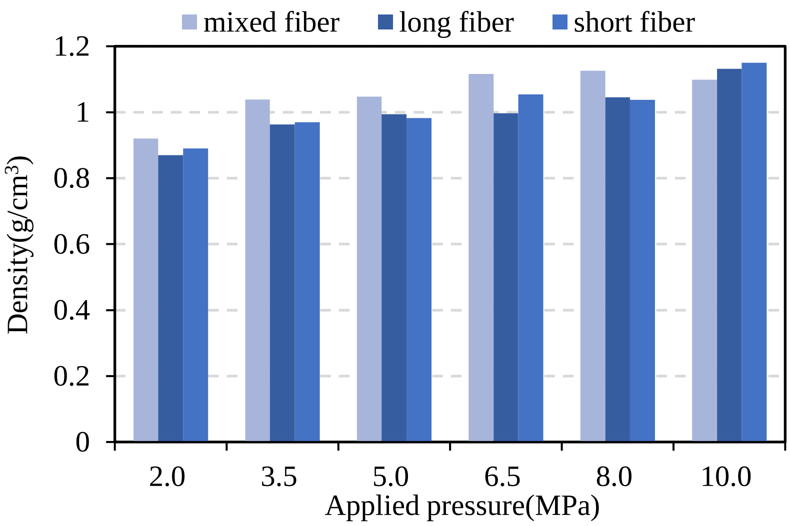


Fig.6-3 The density of bio-board at different fiber lengths.

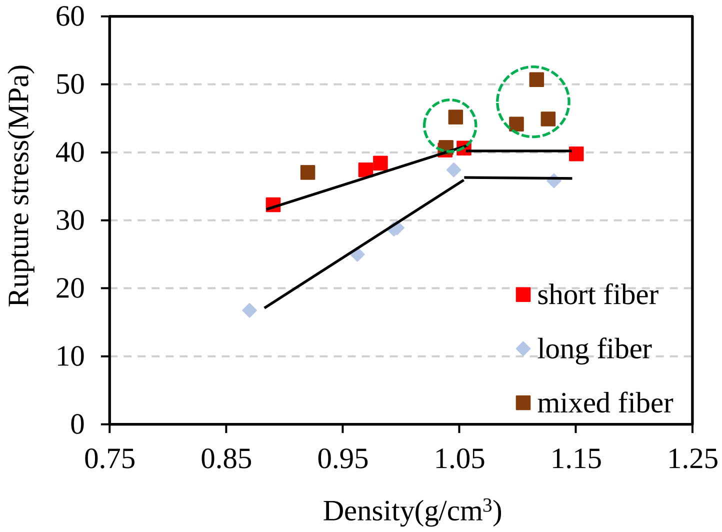


Fig.6-4 Bending rupture stress of bio-board at different fiber lengths.

Fig.6-4 shows the results of bending rupture stress of bio-boards against the density. Both density and fiber length are statistically significant at 99% confidence level for bending rupture stress of bio-board and hence have caused a significant variation in bending properties. The bending rupture stress of bio-board made from long fibers was ranging from 16.8 to 37.4 MPa (increased by 122.6%). The bending rupture stress of bio-board made from short fibers was ranging from 32.3 to 40.6 MPa (increased by 25.7%). The bending rupture stress of bio-board made from mixed fibers was ranging from 37.1 to 50.7 MPa (increased by 36.7%).

The bending rupture stress of short fiber bio-boards are approximately two times higher than those of the long fiber bio-boards at the low density (made at 2 MPa), However, this difference is less significant with the increase of bio-board densities. Besides, both the short fiber bio-board and long fiber bio-board, the bending rupture stress increased linearly, before the density reached near the 1.05 g/cm^3, after that, the bending rupture stress tends to a stable value of 40 MPa for short fiber bio-board and 36 MPa for long fiber bio-board, respectively.

For mixed fiber bio-board, the bending rupture stress reaches 37 MPa when the bio-board made at 2 MPa, which is higher than the bending rupture stress of all the long fiber bio-boards. When the bio-board was manufactured at an applied pressure of 3.5 and 5 MPa, the density of them is very close, but the bending rupture stress differs by 10%. Similar results were also found when the bio-board made at 6.5, 8 and 10 MPa. The bending rupture stress of mixed fiber bio-boards was basically higher than that of short fiber bio-board at equal density level.

Long fibers are more sensitive to changes in applied pressure and require higher applied pressure to obtain better bending performance than those required for short fiber bio-boards. For mixed fiber bio-boards, the bending rupture stress not only affected by the applied pressure but also associated with the composite structure mixed by short fiber and long fiber. The composite structure is conducive to improving bending properties, and have caused a significant variation of bending properties.

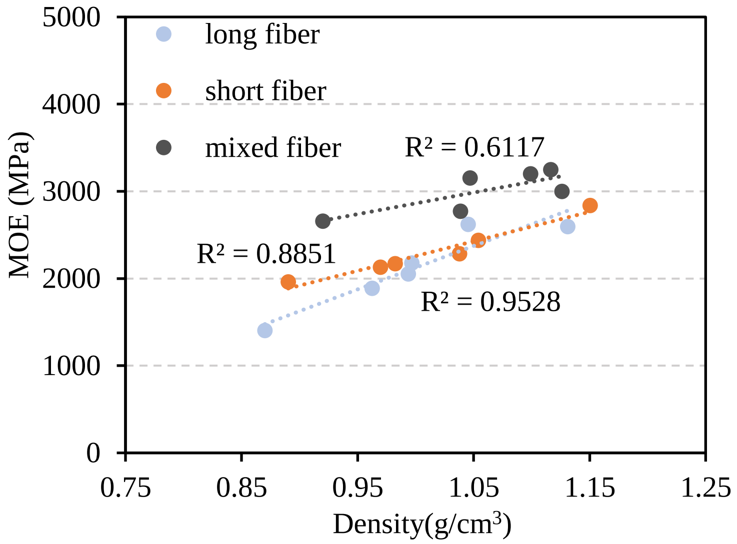


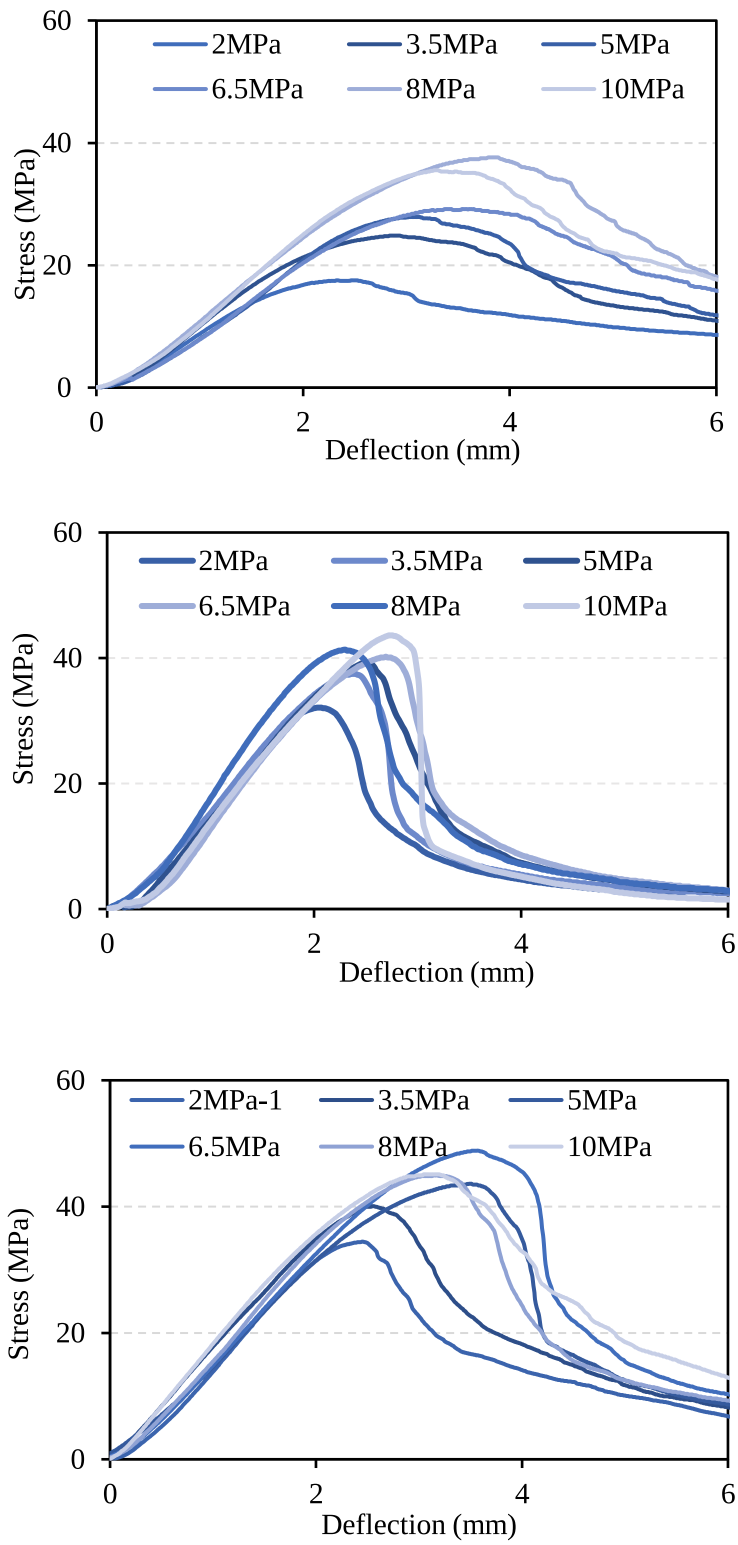
Fig.6-5 Modulus of elasticity of bio-board at different fiber lengths.

Modulus of elasticity (MOE) is plotted as a function of panel density in Fig.6-4. Fiber length is statistically significant at 95% confidence level for MOE of bio-board. The density is statistically significant at 95% confidence level for MOE of short fiber bio-boards and long fiber bio-boards, whereas it is not statistically significant for mixed fiber bio-boards.

The MOE of bio-board made from long fibers was ranging from 1403.3 to 2623.0 MPa (increased by 86.9%). The MOE of bio-board made from short fibers was ranging from 1961.9 to 2839.1 MPa (increased by 44.7%). Both short fiber bio-board and long fiber bio-board, The MOE increased linearly with the rise of the density, When the density is lower than 1.0 g/cm^3, the MOE of the long fiber bio-board is lower than that of the short fiber bio-board, then the MOE of them get close to each other. The MOE of bio-board made from mixed fibers was ranging from 2658.0 to 3248.5 MPa (increased by 22.2%), which was higher than that of two non-mixed fiber bio-board at equal density level, and the fluctuation of MOE was also higher than that of two non-mixed fiber bio-board. This result was closely related to the unique structure combined with the long fiber and short fiber.

As an example, the stress-deflection curve of specimens was selected from every bio-board made at applied pressure of 2 to 10 MPa, and it was shown in Fig.6-6. In Fig.6-6 (a), the peak value of stress-deflection curve of long fiber bio-board was lower than that in Fig.6-6 (b) and (c) at the same applied pressure condition. The specimens were failed at this point reaching the bending rupture stress. In the initial stress phase, the specimen is in the stage of elastic deformation, in which the deflection has a linear relationship with the stress. With the increase of external force, the stress-deflection curve is not linear, indicating that it is an inelastic deformation stage, and the resulting deformation cannot be restored to its original state. Subsequently, the specimen was slowly destroyed, and the failure time was long. The interfacial binding capacity of long fibers is weak, so the specimens cannot form a firm bond in the thickness direction. After the lower layer fails due to the tensile force, the fibers in the upper layer can still bear a certain load, therefore, the destruction appears layer by layer from the lower surface to the upper surface. In Fig.6-6 (b), for the short fiber bio-boards, the stress-deflection curve has a higher slope of the rising stage, after the failure point, the bending stress rapidly dropped to a certain value (around 15MPa) then slowly approached to 0. Bio-boards made of short fibers are firmly combined in the thickness direction as a whole part. When the applied load reaches the specimen' s limit, the whole specimen's body fails instantly, except a few fibers remain connected.

In Fig.6-6 (c), the peak value of stress-deflection curve of mixed fiber bio-board was higher than that of two non-mixed fiber bio-board at the same applied pressure condition. The bio-board made of mixed fibers combines the merits of long fibers and short fibers. The fiber inside the board firmly connected, so the peak value of its stress-deflection curve is higher. Due to the interweaving of long fibers, the destructive appear layer by layer, and the specimens can still withstand greater loads for a longer time when the specimen's failure occurs.



(a) long fiber bio-board; (b) short fiber bio-board; (c) mixed fiber bio-board.

Fig.6-6 Stress-deflection curve of bio-board at different applied pressure.

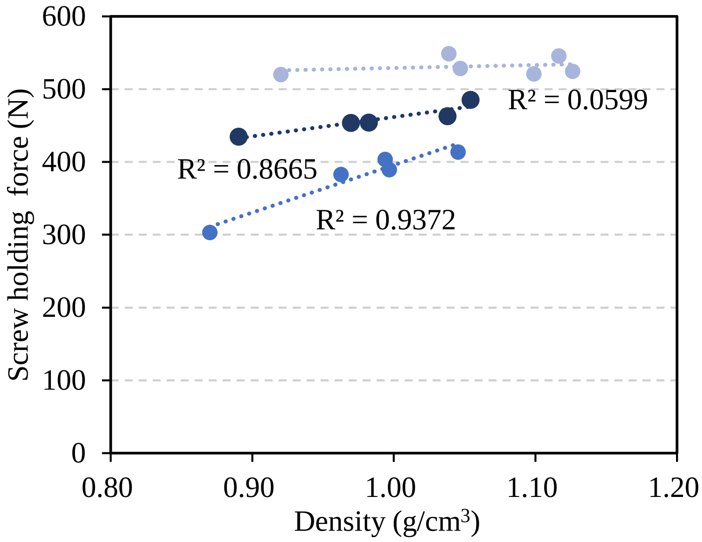


Fig.6-7 Screw holding force of bio-board at different fiber lengths.

Screw holding force (SHF) is plotted as a function of panel density in Fig.6-7. Fiber length is statistically significant at 95% confidence level for SHF of bio-board. The density is statistically significant at 95% confidence level for SHF of short fiber bio-boards and long fiber bio-boards, whereas it is not statistically significant for mixed fiber bio-boards. The SHF of bio-board made from long fibers was ranging from 303.2 to 413.8 N (increased by 36.5%). The SHF of bio-board made from short fibers was ranging from 434.7 to 485.6 N (increased by 11.7%). The SHF of bio-board made from mixed fibers was in a narrow range of 520.2 to 548.8 N.

Generally, the SHF value of mixed fiber bio-board was higher than that of short fiber bio-board and which was higher than that of long fiber bio-board, in turn. Both the long fiber bio-board and the short fiber bio-board, the SHF value of them was increased linearly with the rise of density, whereas the effect of density change is more profound for long fiber bio-board than that for short fiber bio-board. For mixed fiber bio-board, the SHF of it is barely changed with the variation of density.

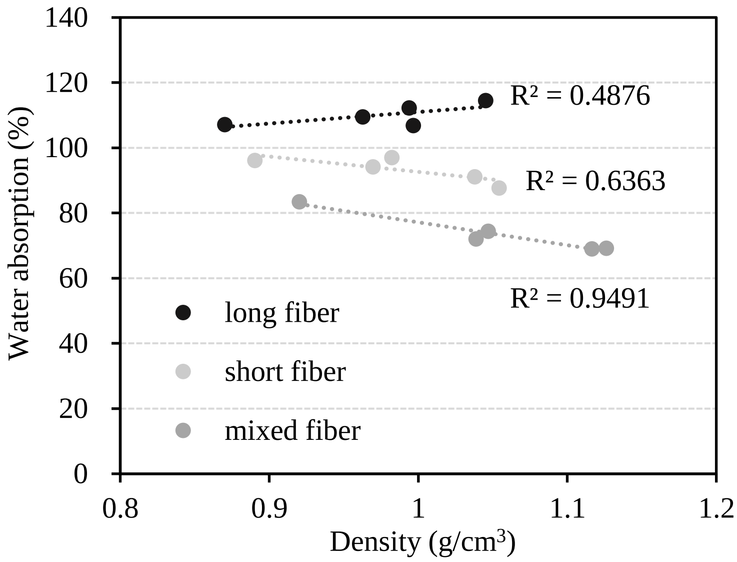


Fig.6-8 Water absorption of bio-board at different fiber lengths.

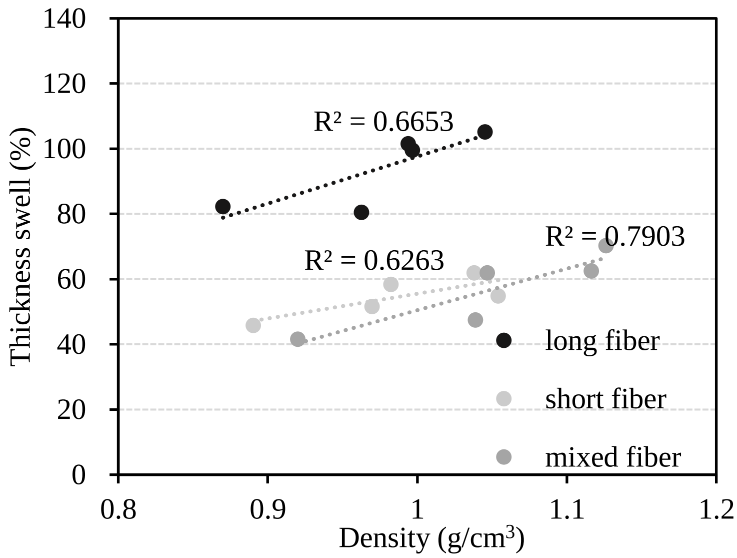


Fig.6-9 Thickness swell of bio-board at different fiber lengths.

The TS and WA result in Fig. 6-8 and Fig. 6-9 reveal that both fiber length and density have influenced dimensional properties. The average WA of short fiber bio-boards and mixed fiber bio-boards decrease from approximately 96.1% to 87.7% and 83.5% to 69.3% when the bio-board density is increased from 0.9 to 1.1 g/cm^3, respectively. For long fiber bio-boards the value is in the range of 106.9% to 114.5% without pronounced trend. But, the average TS of bio-boards shows a opposite trend compared to the WA, that is, the TS value increase with the rise of bio-board density all of three kinds of fiber length.

The results in this study show that since the high-density bio-board have lower permeability and consequently a smaller number of voids, the water absorption properties are significantly decreased. However, high density bio-board were compacted under higher pressures, when exposed to water, the stresses induced during hot pressing are released which causes a pronounced reversal of panel densification, resulting in comparatively high TS. A similar phenomenon was observed by Abdul Khalil et al.

The mixed fiber bio-boards have shown conceivably better dimensional properties than long and short fiber length bio-boards. Since short fibers are stuffed among the long fibers, which were conducive to form a dense structure causes smaller voids and lesser pathways for water penetration. As a result, comparatively less free water would be available for weakening the fiber bonds. This has resulted lower TS and WA for mixed fiber bio-boards as compared to long fiber and short fiber bio-boards.

6.4 Summary

In this chapter, the influence of fiber length and density on mechanical properties and dimensional stability performance of bio-board was investigated. Some conclusions were revealed as follows:

(1) Generally, the high-density bio-board have shown excellent mechanical properties. As for dimensional stability, the effect of density on TS and WA is inconsistent. High-density bio-boards have lower water absorption properties but higher thickness swell properties.

(2) The properties of bio-boards closely affected by the fiber length. The mixed fiber bio-boards have shown conceivably better mechanical properties and dimensional properties than long and short fiber length bio-boards, due to its dense structure in which short fibers are stuffed among the interweaving of long fibers.

Chapter 7. The soybean straw-polylactide based hybrid bio-board and its properties

7.1 Introduction

The above chapters discussed the effects of manufacturing conditions and fiber morphology on the mechanical properties and dimensional stability of the bio-board. The manufactured bio-board had good mechanical properties and could meet the requirements of the specific standard of JIS A5905. However, these bio-boards generally had a poor water-proof performance, which could not meet the standard of JIS A5905. Hence, this chapter mainly discusses the methods to improve the water-resistance properties of bio-board.

Most traditional fiberboards are added with synthetic resin to obtain excellent mechanical properties and water resistance. This manufacturing process has two major drawbacks. First, they contain volatile organic compounds harmful to human health. Second, the petroleum feed stocks for producing formaldehyde are limited. In some recent studies researchers have investigated the alternative candidates of synthetic resins for board manufacturing industry, e.g., soy protein adhesives, starch-based wood adhesive, tannin, lignin, glucose, sucrose, biological wax[62][66]. Most of the research on the application of these formaldehyde-free wood adhesives to the production of fiberboards was in the laboratory or small-scaled stage. The addition of them in fiberboard production could increase dimensional stability to some extent, but it may also reduce the mechanical properties.

Recently, there have been reports on wood-plastic composite materials (WPC). The WPC is a composite product, in which polyethylene, polypropylene, polylactic acid (PLA), polyhydroxyalkanoates (PHA) was used to replace common resin adhesives, mixed with more than 50% of wood or agricultural plant fibers, and further experiencing extrusion, hot-pressing or injection molding. Yao et al.[67] manufactured composite board using HDPE (high density polyethylene) and natural fibers by melt compounding and compression molding. The study showed that rice straw fibers can work well with HDPE as reinforcing filler. The extrusion method was applied to prepare composites with peanut husk, rice husk and walnut shell based on HDPE, and the results found that the rice husk/HDPE composite had the best mechanical strength and creep resistance[68]. Zou et al., made lightweight composites wheat straw and polypropylene (PP) webs, the properties of which was superior to jute-PP composites with the same density[69]. A similar research was also reported by Yemele et al[70]. and Jayamani, et al[71].

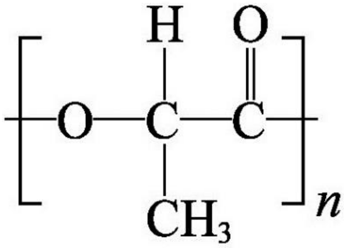
More lately, degradable plastics such as PLA and PHA are also used in the production of WPC. This new class of composites is more environmental-friendly due to the lower environmental impact of both natural fibers and PLA resin during their production and usage lifetime as well as at their end of life [72]. Different types of natural fibers and fillers have been compounded with PLA, including recycled wood [73], bamboo[74], flax[75]. Most of these studies showed that it is possible to improve the mechanical properties and dimensional stability to a certain degree when using PLA to create WPC.

Moreover, further treatments such as coating or laminates ware also discussed by some researchers. Rhim et al.[76][77] produced PLA-coated paperboards (PB) and evaluated the water barrier properties, including water vapor permeability (VWP), water absorptiveness (WA), and contact angle (CA) of the water drop. They found that water resistance of paperboard was improved through surface coating with PLA. Mondala et al.[78] applied patent in which the chitosan as a biobased barrier coating for functional paperboard products.

The PLA is commercially available with good performance as biodegradable packaging material, which is derived from renewable resources, such as corn or sugar beets. In addition, PLA is a thermoplastic material of comparable mechanical performance to petroleum-based polyesters, especially high elasticity modulus and stiffness. In this chapter, the soybean straw/PLA based hybrid bio-board and coating bio-board were manufactured, respectively. For hybrid bio-board, the PLA mass percentage on bending properties and water-proof performance were investigated of the bio-board made at different applied pressure. For coating bio-board, the coating method was discussed, and the effect of PLA coating on bending properties and water-proof performance was tested, as well.

7.2 Materials and method

The commercially available polylactic acid (4032D, Nature Works Co., Ltd. USA) were purchased and ground into powder. The PLA had a density of 1.25 g/cm^3, a molecular weight (Mw) between 100,000 and 200,000 g/mol, tensile modulus of 3 GPa, tensile strength of 50-70 MPa, flexural modulus of 5 GPa, flexural strength of 100 MPa, and an elongation at break of about 4%. They showed a glass transition temperature (T_g) of 55-60 °C and a melting temperature (T_m) of 170-180 °C.



PLA

Fig.7-1 PLA used in this study.

Table 10 The making condition of hybrid bio-board.

No.	PLA mass percentage（%）	Applied pressure (MPa)	Forming temperature (℃)	Forming time(min)	Melting temperature (℃)	Melting time(min)
1		2				
2	10	5				
3		8				
4		2				
5	30	5	160	30	180	10
6		8				
7		2				
8	50	5				
9		8				

The preparation of the soybean straw fiber and PLA hybrid bio-board was performed utilizing the traditional hot-press manufacturing process. The soybean straw fiber, PLA were mixed according to the mass percentages listed in Table 7-1 and then well-stirred for 5-8 min. The blended fibers were poured into the mold and pre-formed at heating temperature of 160 ℃ for 30 min by using hot-press. After that, the pre-formed hybrid bio-board was placed between the parallel flat metal plates at heating temperature of 180 ℃ for 10 min to make the PLA inside board fully melted.

The surface of the hybrid board after the forming process is rougher than that of the pure bio-board since PLA on the surface of the board were not fully melted. Besides, the PLA particles stuck to the aluminum mesh and torn off when demolding, resulting in small pits on the surface. After the second time melting process, the surface of hybrid bio-board becomes smoother, and the color of it is darker. The produced hybrid bio-boards are shown in Fig. 7-2.

Fig.7-2 Surface and cross-section of produced hybrid bio-board

The preparation steps of the coating bio board are as follows: Firstly, pure bio-board was produced under the conditions of table 12. The applied pressure was 2 MPa, 5 MPa, and 8 MPa. The condition of heating temperature was 160 ℃, and the forming time was 30 min.

Then, the produced bio-board was cut into 5 pieces of bending test specimens and 2 pieces of water absorption test specimens. Finally, the melted pure PLA was applied evenly on the surface and section of bio-board specimens, and the roller was used to make the coated PLA layer uniformly and flat distributed.

Table 11 The making condition of pure bio-board for coating.

No.	Heating temperature (°C)	Applied pressure (MPa)	Forming time (min)
A	160	2.0	30
B	160	5.0	30
C	160	8.0	30

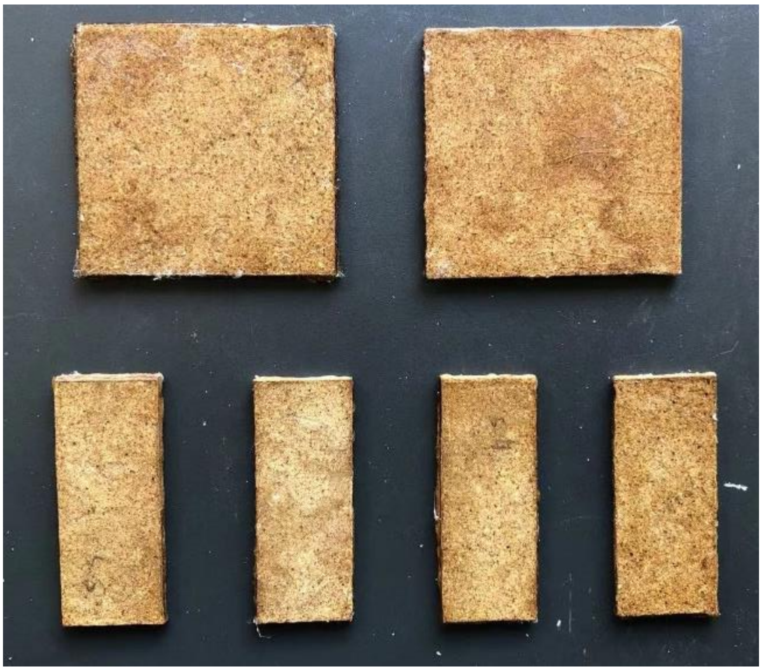


Fig.7-3 Prepared coating bio-board specimens

7.3 Results and discussion

As shown in Fig.7-4, the density of hybrid bio-board made at 5 MPa and 8 MPa was almost same as 1.0 g/cm^3 under different mass percentage of PLA, whereas the density of hybrid bio-board made at 2 MPa was slightly increased from 0.92 to 0.97 g/cm^3 with the increase of PLA mass percentage. The density of hybrid was not only affected by the making condition but also associated with the density of PLA and soybean straw. When the board made at 2MPa, the density was mainly affected by the fact that the density of pure PLA (1.25

g/cm^3) is higher than the soybean straw fiber. The moisture content of hybrid bio-board is shown in Fig.7-5. The moisture content was decreased with the increase of PLA mass percentage, which is attributed to the hydrophobicity of the PLA.

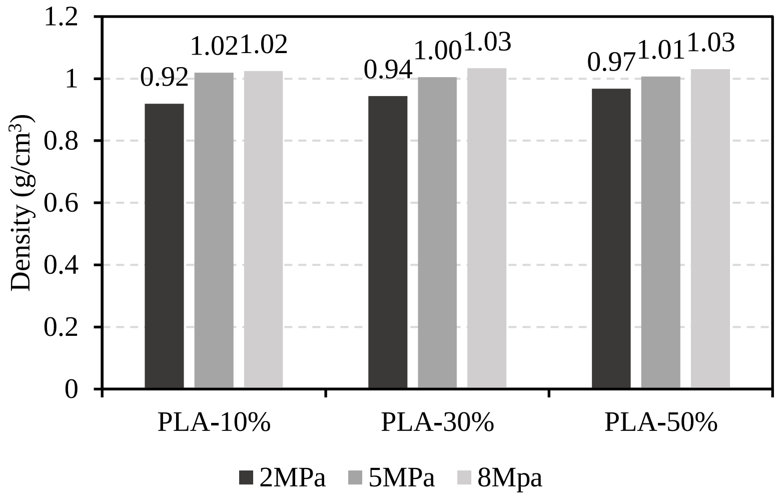


Fig.7-4 Effect of PLA mass ratio on density of hybrid bio-board.

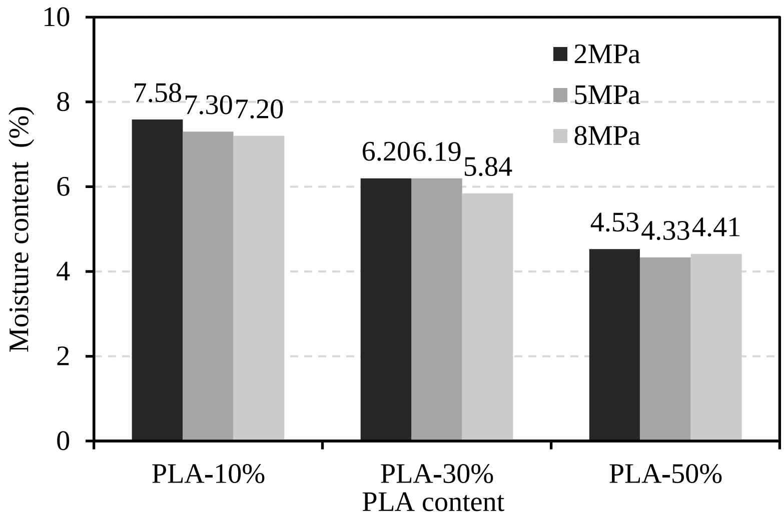
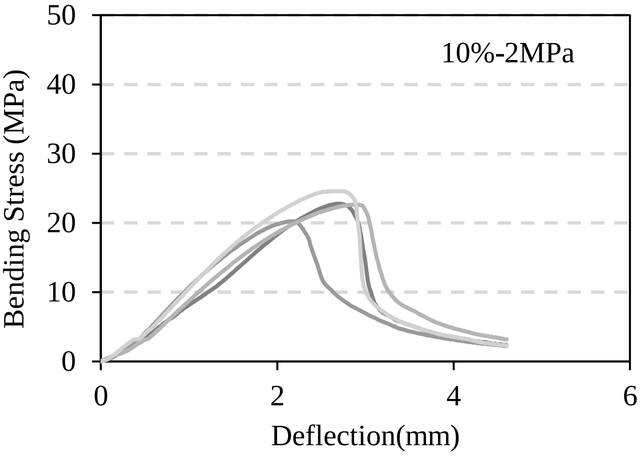
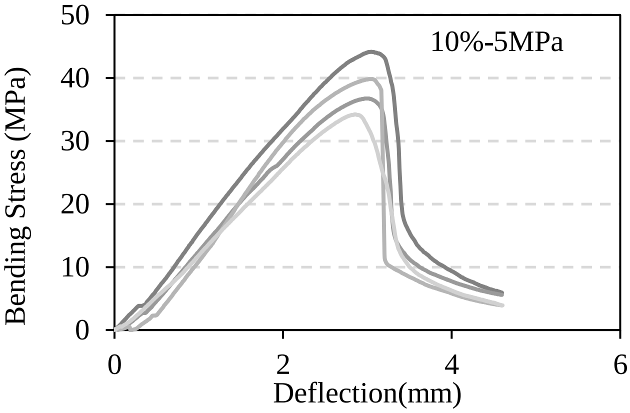
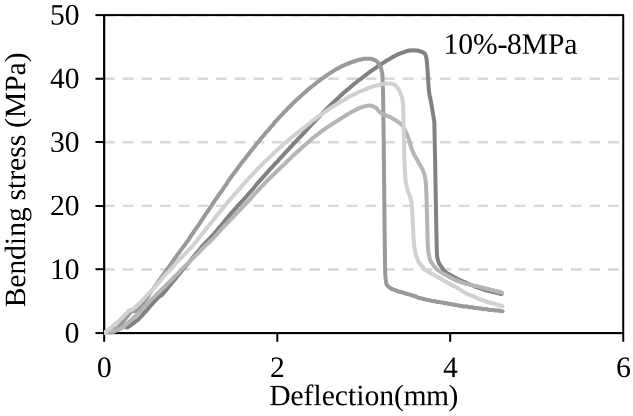


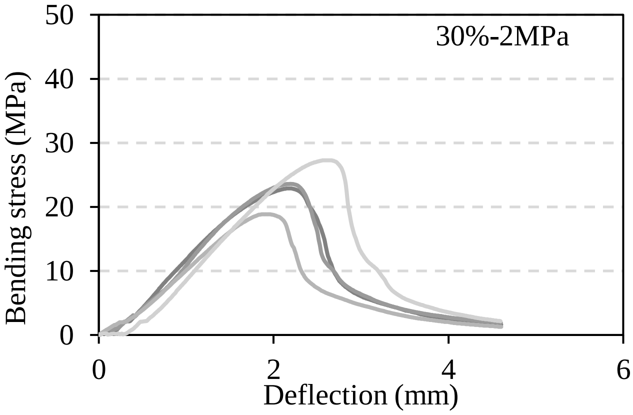
Fig.7-5 Effect of PLA mass ratio on moisture content of hybrid bio-board

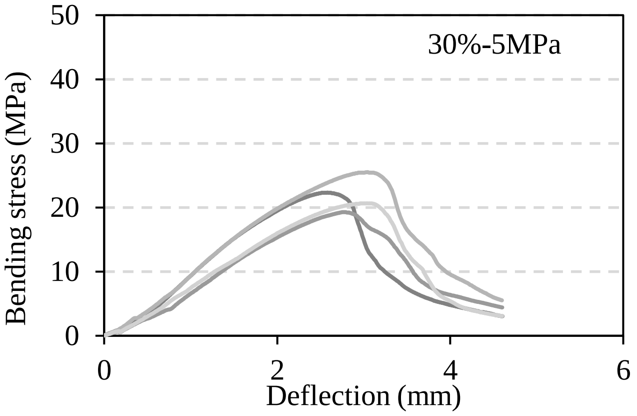
As shown in Fig.7-6, all the bending stress-deflection curve of hybrid bio-board almost approach a straight line before the fractures occurred. When the failure happened, in most cases, the bending stress straightly drops to a particular value, and then slowly approaches to zero, but in Fig.7-6 (a), (d), (e), and(g),the curve declines with a slope, because the boards in these cases were made in low applied pressure. These results were also related to the aggregation state of fiber and PLA inside the board.

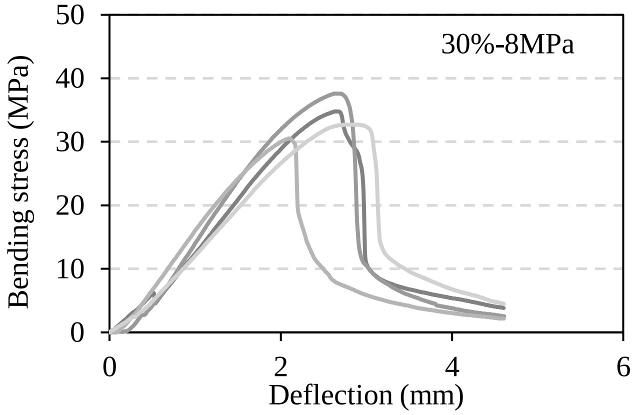

10%-2MPa
Bending Stress (MPa)
Deflection(mm)
0
10
20
30
40
50
0
2
4
6

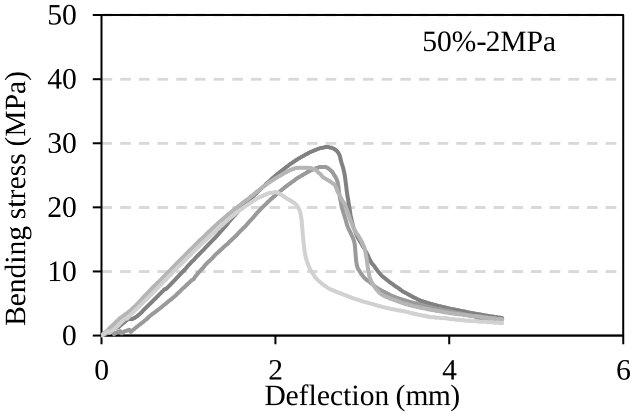

10%-5MPa
Bending Stress (MPa)
Deflection(mm)
0
10
20
30
40
50
0
2
4
6

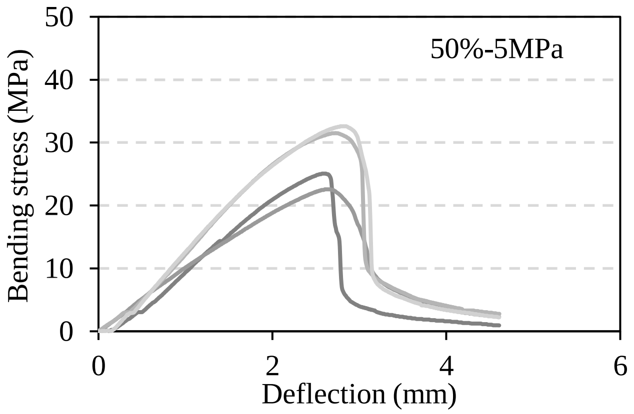

10%-8MPa
Bending stress (MPa)
Deflection(mm)
0
10
20
30
40
50
0
2
4
6


30%-2MPa
Bending stress (MPa)
Deflection (mm)
0
10
20
30
40
50
0
2
4
6


30%-5MPa
Bending stress (MPa)
0
10
20
30
40
50
0
2
4
6
Deflection (mm)


30%-8MPa
Bending stress (MPa)
0
10
20
30
40
50
0
2
4
6
Deflection (mm)


50%-2MPa
Bending stress (MPa)
0
10
20
30
40
50
0
2
4
6
Deflection (mm)


50%-5MPa
Bending stress (MPa)
0
10
20
30
40
50
0
2
4
6
Deflection (mm)

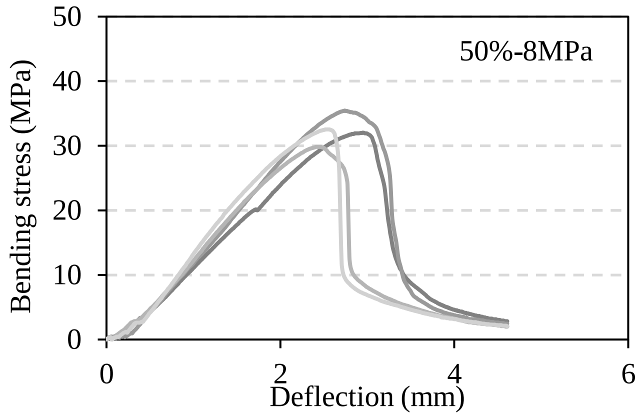


Fig.7- 6 Bending stress-deflection curve of hybrid bio-board.

As shown in Fig.7-7, the bending rupture stress of hybrid bio-board using 10% of PLA was higher than that of other PLA mass percentage, and the value of them has no apparent difference compared with bending rupture stress of pure bio-board without PLA. The bending rupture stress value of hybrid bio-board by using 30% of PLA and 50% of PLA were almost similar slightly increased from 24 to 32 MPa with the increase of applied pressure. The bending properties of the hybrid bio-board with a higher percentage of PLA were not only lower than the pure bio-board but also lower than the flexural strength of PLA used in this study.

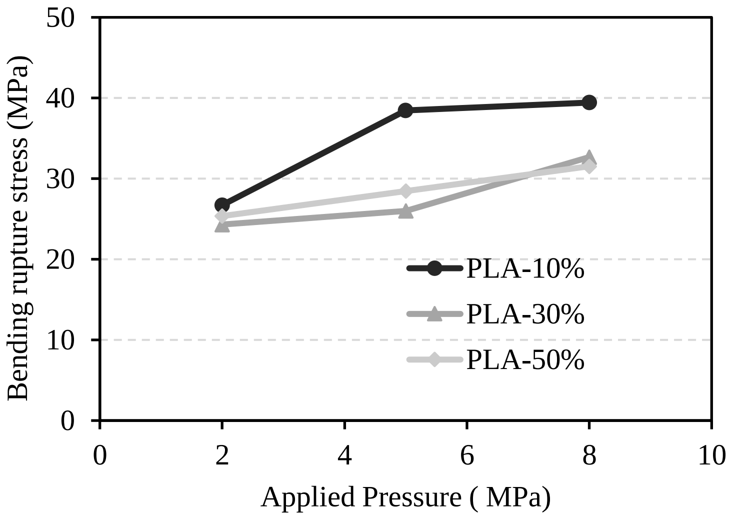


Fig.7-7 Effect of PLA mass ratio on bending rupture stress of hybrid bio-board.

This result might be caused by the following three reasons. First, the fiber and the PLA could not well mixed in the water since the density of PLA is higher than straw fiber. Secondly, the PLA inside board might not be fully melted, and which affected the bonding quality of bio-board. Thirdly, biomass straw is hydrophilic due to the presence of hydroxyl groups, but the polylactic acid is hydrophobic. Polar hydrophilic biomass straws have poor compatibility with non-polar hydrophobic PLA, leading to the interface strength of straw and PLA is un-tightly. There is an effective method to increase the interfacial bonding force by adding the coupling agent reported by Nikrai et al., and Simão et al[79][80].

The results of WA and TS are plotted in as a function of applied pressure in Fig.7-8 and Fig.7-9. Both WA and TS decreased with the increase of PLA mass percentage, which is attributed to the hydrophobicity of the PLA. The TS of hybrid bio-board made with 10% of PLA mass percentage was increased from 22.3% to 28.7%, when the applied pressure increased from 2 to 8MPa, and which was similar to the trend of TS of pure bio-board discussed in cheaters above. The TS of hybrid bio-board made with 50% of PLA mass percentage was in a narrow range of 12.1% to 13.7%, and less sensitive with the increase of applied pressure. The WA value of hybrid bio-boards made of different proportions of PLA tends to decrease, with the increase of pressure. As described in JIS A5908, the WA of hardboard S35 type should not be greater than 35%. Therefore, when the making condition of applied pressure is higher than 5 MPa, and the PLA mass percentage is above 30%, the produced bio-board could meet the JIS standard for this parameter.

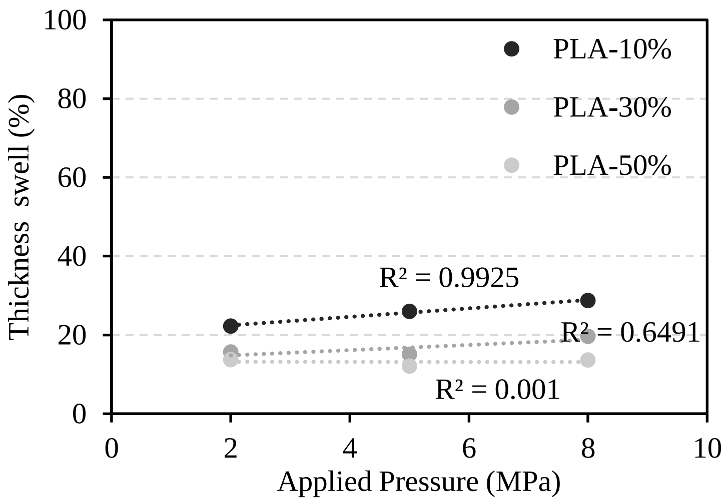


Fig.7-8 Effect of PLA mass ratio on TS of hybrid bio-board.

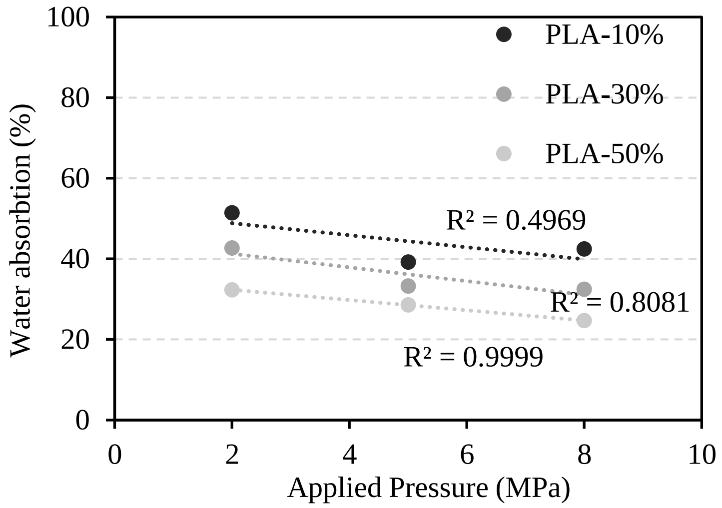


Fig.7-9 Effect of PLA mass ratio on WA of hybrid bio-board.

As shown in Fig.7-10, the bending rupture stress of coating bio-board was generally higher than the non-coating bio-board. Especially, the bending rupture stress of coating bio-board made at 2 MPa was more than two times of non-coating bio-board. Besides, these values were no significant differences under different applied pressure. The following two reasons might cause the improvement of bending properties. First, the bending properties of PLA itself were higher than those of pure fibers; Secondly, after the pure board was coated with PLA, an outer hard and inner tough structure was formed, which was also conducive to the board to withstand higher bending load. Therefore, the amount of PLA applied to the specimens also affected the bending properties.

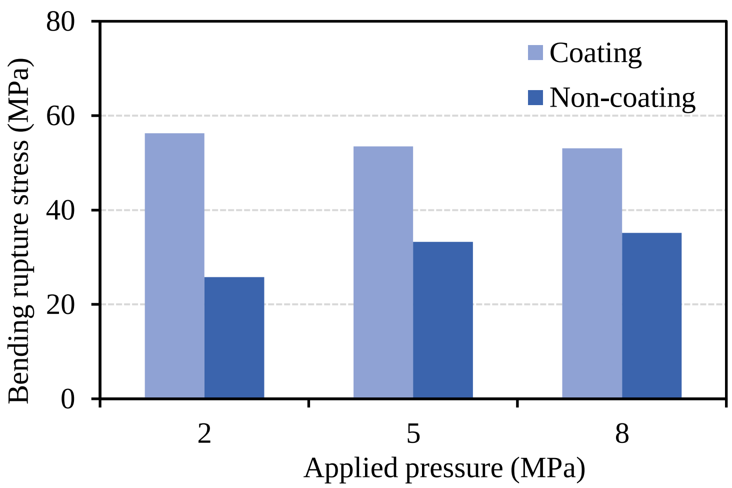


Fig.7- 10 Bending rupture stress of coating bio-board

As shown in Fig.7-11, all the bending stress-deflection curve of coating bio-board in a straight line before the fractures occurred. When the failure happened, the bending stress straightly drops to a small value, and then slowly approaches to zero, but in some cases, the bending stress has a slight rebound. This phenomenon might be due to the fact that the PLA coating on one side is broken immediately, but the fibers in the middle layer are still connected, and it has a certain elasticity.

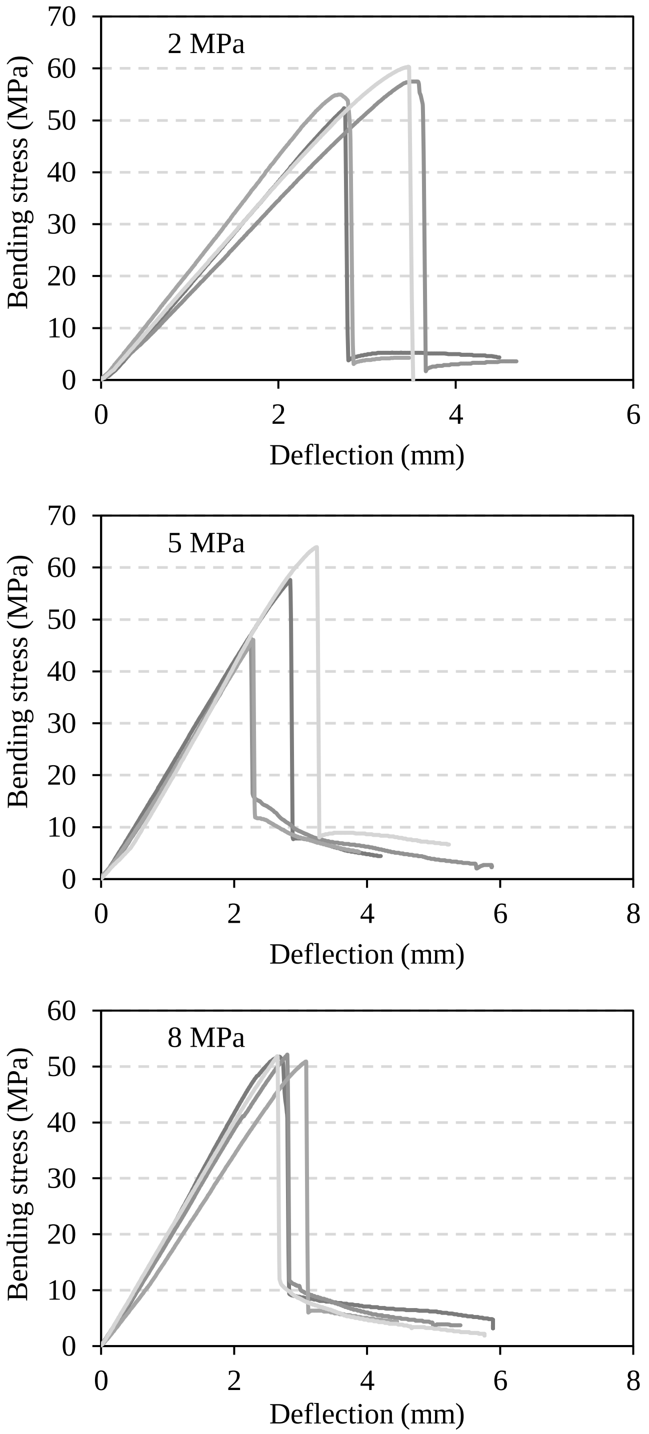


Fig.7- 11Bending stress-deflection curve of coating bio-board

The results of WA and TS of coating bio-board are shown in Fig.7-12 and 7-13. The WA of coating bio-boards was ranging from approximately 36.04% to 25.81%, and the TS of them was ranging from 19.67% to 25.50%. Under the same conditions, the WA of non-coating bio-boards were between 75.72% and 83.80%, and the TS of them were between 43.73% and 56.35%. The WA and TS of coating bio-board have dropped significantly, which is smaller than 1/2 of non-coating bio-board. Because the PLA coating is crispy after cooling, the fissure will inevitably occur. When the test piece was immersed in water for a long time, water will penetrate the test piece through the fissure. Then the fiber will absorb water and expand, which makes the fissure enlarged, and the water will further penetrate the test piece. As a result, these values have not dropped to the expected 0.

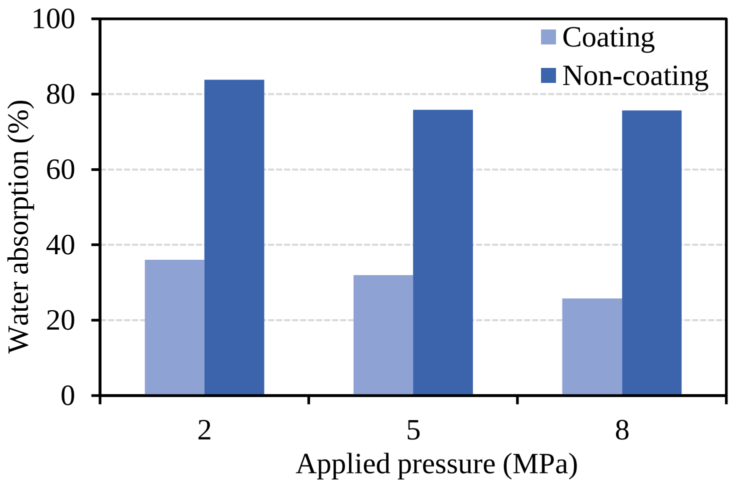


Fig.7- 12 Water absorption of coating bio-board

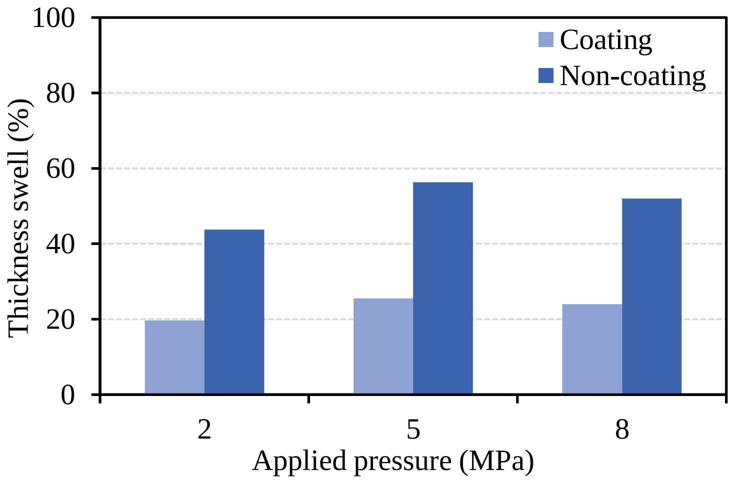


Fig.7- 13 Thickness swell of coating bio-board

7.4 Summary

In this chapter, the soybean straw-PLA based hybrid bio-board and coating bio-board were made at different applied pressure, and the properties of these bio-boards were evaluated. For the hybrid bio-board, the influence of PLA mass percentage on mechanical properties and dimensional stability performance of bio-board was also investigated. Some conclusions were revealed as follows:

(1) The bending properties of the hybrid with 10% of PLA was comparable with pure bio-board, but the water resistance properties were highly improved. With the increase of PLA mass percentage, the bending properties were unexpected decreased, but the water-resistance properties were much more improved.

(2) Coating method was a valid method. Since the coating bio-boards have shown conceivably better bending properties and water-resistance properties than pure bio-board.

Chapter 8. Conclusions and future work

8.1 Conclusions

The purpose of this research is to manufacture bio-board by using soybean straws which consist of a large amount of lignin, with performance comparable to commercial standards. The physic-mechanical properties of soybean straw bio-board have been determined by standard procedures based on JIS A5905. The influence of applied pressure, forming time, and heating temperature have been investigated to get the excellent properties. For the effect of forming time, the properties of corn straw bio-board were also evaluated. The fiber length and board density on mechanical properties and dimensional stabilities on bio-board was also studied. Two methods were tried to improve the waterproof of the bio-board. Bio-board with excellent mechanical properties have been successfully manufactured. The specific conclusions from the work are summarized in the following sub-sections:

(1) The results indicate that the bio-boards were successfully made and has certain strength. thereby, the making process proposed in this research is valid way to produce board.

(2) The density increased with the increase of applied pressure, whereas, the moisture content shows the opposite tendency. Mechanical properties of bio-board, such as bending properties, tensile properties, and screw holding force, were closely related to the density and moisture content affected by the applied pressure.

(3) The TS value of bio-board increased with the increase of applied pressure, and the WA of them was over 85%, overall, bio-board has poor water-proof properties, and the bending performance is also significantly decreased after absorbing water.

(4) Except for the bio-board made at 0.5 h that has relatively low strength and water-proof performance, the performance at other forming time condition did not have significant difference both soybean straw and corn straw bio-board.

(5) The bio-board made of corn straw had better performance in resisting the permeability of water than soybean straw bio-board. The fluctuation of bending rupture stress for corn straw bio-boards was more significant than that for soybean straw bio-board, and the tensile rupture stress for corn straw bio-board were mostly higher than that for

soybean straw bio-board. The properties of the two kinds of bio-boards were closely related to the chemical compositions of raw materials.

(6) In general, with the increase in temperature, there was a decrease in moisture content, the softening of lignin, and the pyrolysis of hemicellulose, which is beneficial to the improvement of the mechanical properties of the bio-board. However, the excessive heating temperature, especially at 230 °C, does not significantly promote improvement to most mechanical properties. On the other hand, the dimensional stability of the bio-board is greatly improved from 140 to 230 °C.

(7) Generally, the high-density bio-board have shown excellent mechanical properties. As for dimensional stability, the effect of density on TS and WA is inconsistent. High-density bio-boards have lower water absorption properties but higher thickness swell properties.

(8) The mixed fiber bio-boards have shown conceivably better mechanical properties and dimensional properties than long and short fiber length bio-boards， due to its dense structure.

(9) The bending properties of the hybrid with 10% of PLA was comparable with pure bio-board, but the water resistance properties were highly improved. With the increase of PLA mass percentage, the bending properties were unexpected decreased, but the water-resistance properties were much more improved.

(10) Coating method was a valid method. Since the coating bio-boards have shown conceivably better bending properties and water-resistance properties than pure bio-board.

8.2 Recommendations for future work

A series of experiments and investigations were carried out to fulfil the three major objectives in this study, and some practical conclusions were summarized as above. However, there are still some research directions or areas that have not be studied or need further study. The following is the recommendations of future works:

(1) The raw materials used in this study was the whole part of soybean straw which sourced from Mie prefecture. The properties of soybean straw are closely related to the planting locations, seasons, and the varieties of seeds. This might influence the results

concluded in this study. Therefore, the soybean straws of different origins and varieties can be selected for further research.

(2) Soybean straw is mainly composed of stems, leaves, and pods. The different parts have different contents of chemical components which could affect the properties of products. So, the next step, the stems, leaves, and pods of soybean straw could be separated, and then make bio-board with the specific part.

(3) Although this study considers the influence of temperature, pressure, and time production conditions on the performance of the bio-board, there is no orthogonal test for these three factors. The next step, orthogonal experiments could be considered and designed to obtain optimal production conditions.

(4) In this paper, powdered polylactic acid is used as an additive to increase the water-resistance of the board. Several other materials, such as PHA, chitosan can also be explored for their potential use in bio-board production. Besides, filmed or fibers of degradable plastic can also be mixed with plant fibers to make composite panels.

(5) The economic viability of bio-board manufacturing with soybean straw fibers should also be assessed.

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Achievements

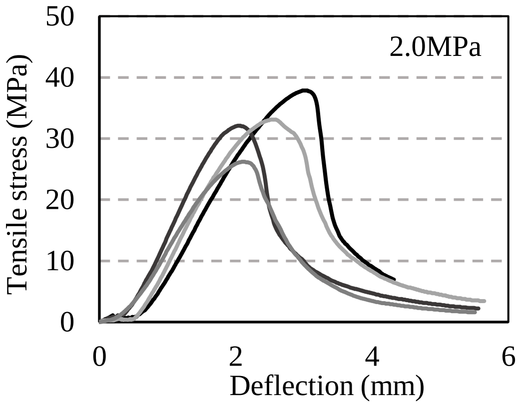
Journal publications:

1) Xiaowen Song and Xiulun Wang: Effects of Pressure on the Mechanical Properties of Bio-board Manufactured by Using Soybean Straw. INFORMATION, Vol.22, No.3, pp.255-262, May 2019.
2) Xiaowen Song, Xiulun Wang and Koji Kito: Effects of Heating Temperature on the Properties of Bio-Board Manufactured by Using Soybean Straw. Materials 2020, 13, 662; doi:10.3390/ma13030662
3) Xiaowen Song, Xiulun Wang and Koji Kito: Effects of Forming Time on the Properties of Bio-board Manufactured by Soybean Straw and Corn Straw, International Agricultural Engineering Journal,

Conference Publications:

1) Xiaowen Song, Xiulun Wang and Koji Kito : Mechanical Properties and Manufacturing Process of Bio-board Using soybean straw, 農業環境工学合同年次大会,愛媛大学,松山市, 2018 年 9 月.
2) Xiaowen Song, Xiulun Wang and Koji Kito : Properties of Bio-board Manufactured by Using Soybean Straw, International Joint Conference on JSAM,SASJ and 13th CIGR VI Technical Symposium Joining FWFNWG and FSWG Workshops, 2019 年 9 月, 北海道大学,札幌市.

Appendix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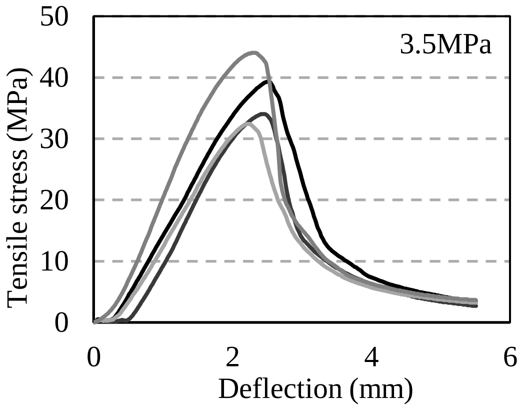
20
30
40
50
0
2
4
6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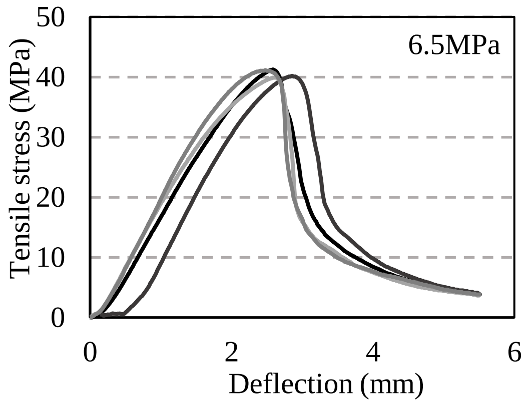
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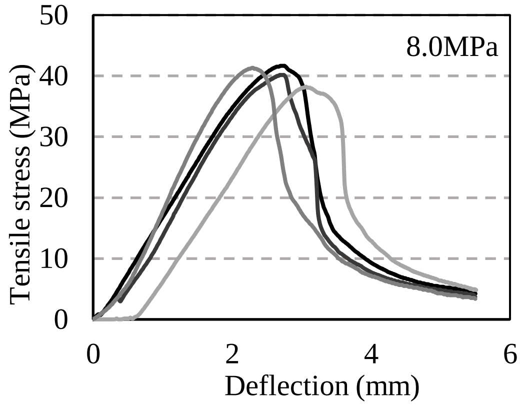
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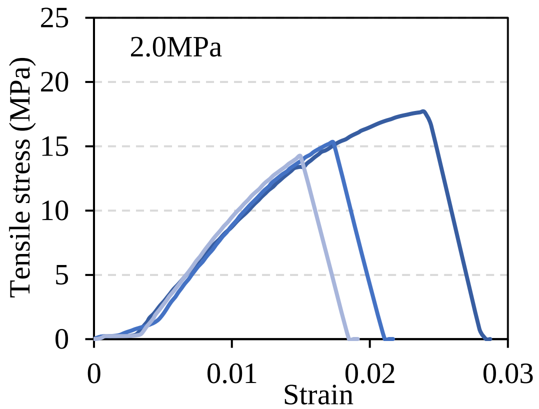
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Strain
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15
20
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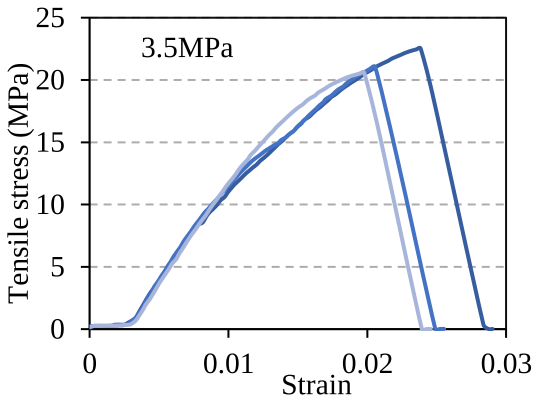
0.01
0.02
0.03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Strain
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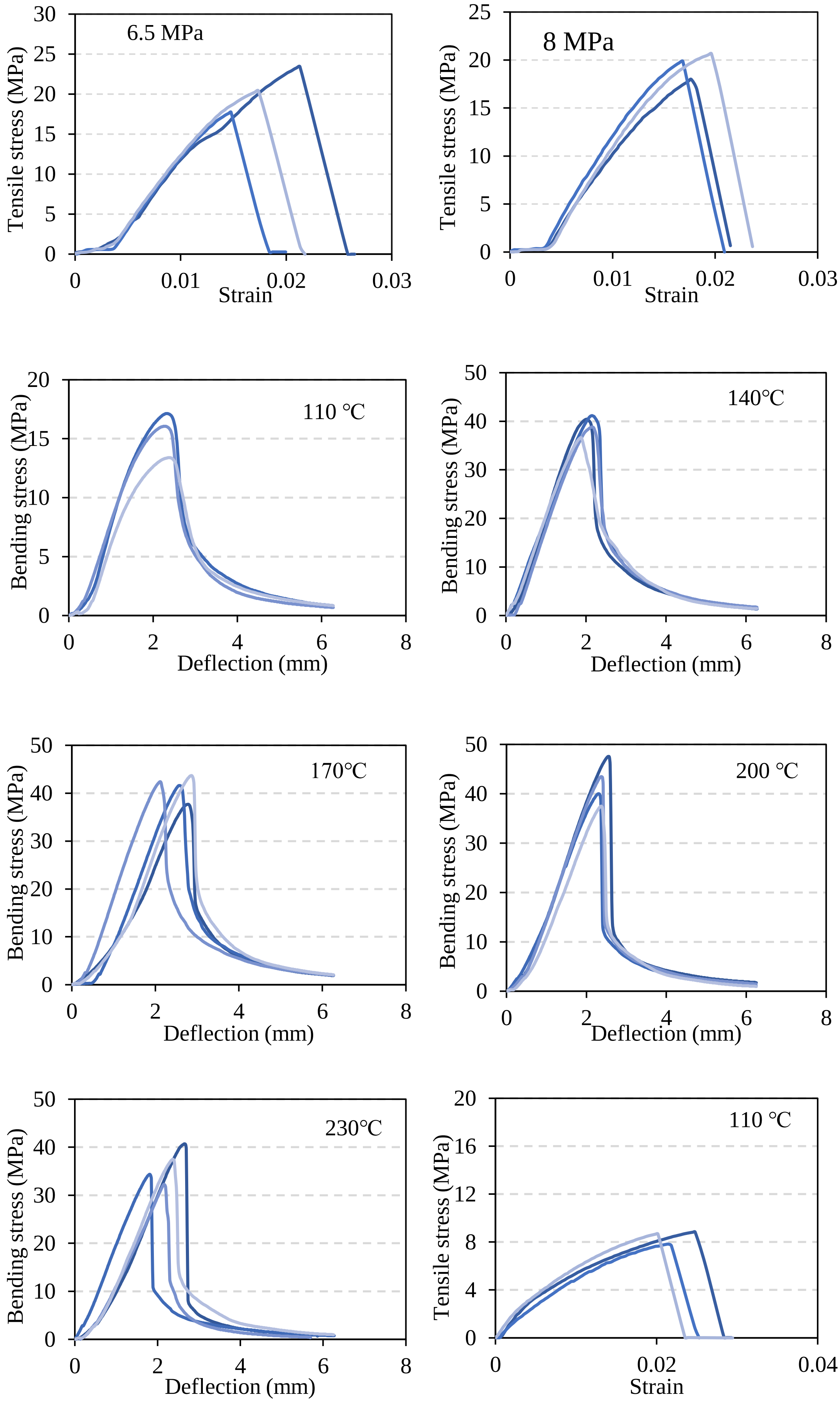
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Strain
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Tensile stress (MPa)
Strain
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Bending stress (MPa)
Deflection (mm)
140°C
Bending stress (MPa)
Deflection (mm)
170°C
Bending stress (MPa)
Deflection (mm)
200 °C
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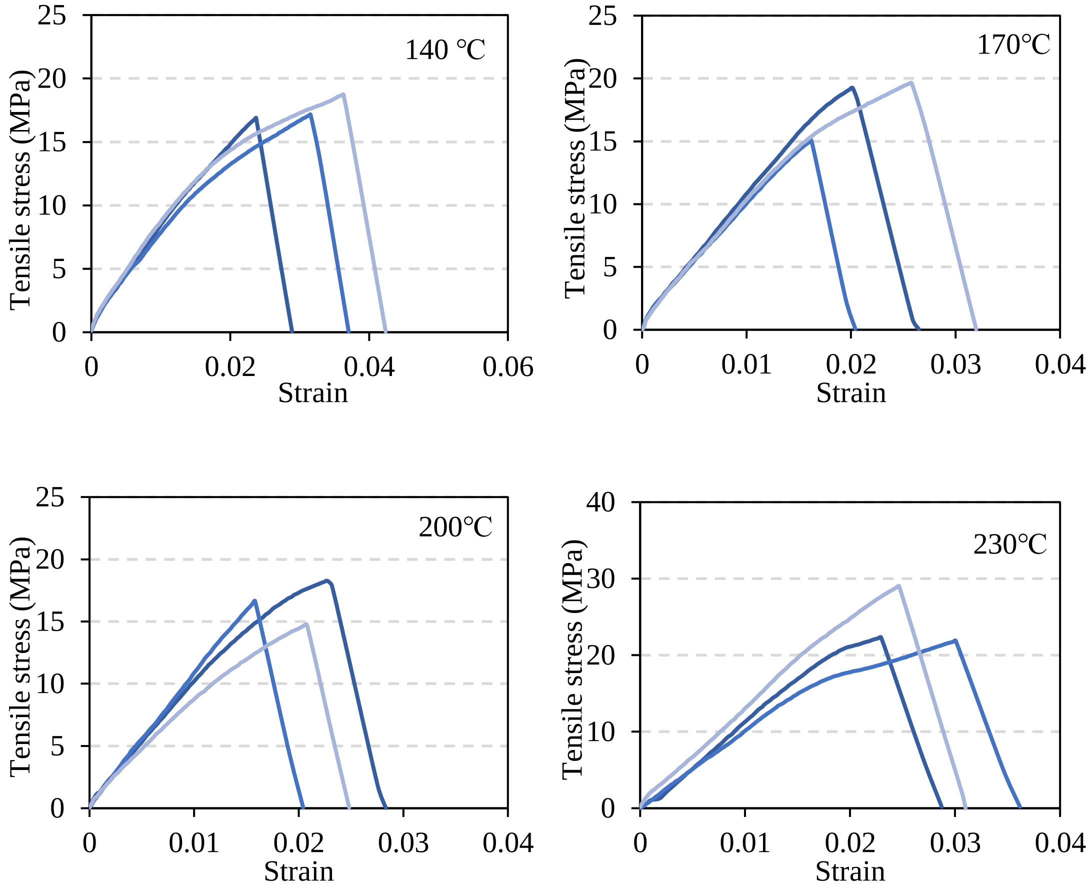
Tensile stress (MPa)
Strain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230℃
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