Ph.D. Thesis

PRODUCTION OF BIODEGRADABLE BOARD AND ITS MECHANICAL PROPERTIES USING CORN STRAW

（トウモロコシ藁を用いた生分解可能なバイオボードの作製及びその強度）

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PREFACE

In order to keep the balance of carbon cycling and make the sustainable development of recycling economy adequately, the low-carbon globalization has been an objective tendency in recent years. One destination of this low-carbon action is to fully utilize the existing biomass resource to produce a new friendly environmental material called bio-board. Furthermore, products made by biomass materials will not bring any environment pollutions after they were used. Bio-boards are also decomposed by microorganism then changed into nutrient substance for vegetative growth.

In this study, corn straw as one of the representative agricultural residues, is mainly used as an experimental raw material. Firstly the background of world utilizations of corn and corn straw is introduced in chapter 1. Basic knowledge of biomass including cellulosic plants was mentioned. Also, other researches which were about biomass material for the past few years were cited and used for reference.

Then, in chapter 2 it is going to develop and optimize the bio-board process to make a bio-board. In board production experiment, two experimental conditions were applied for making two different bio-boards. After the investigation of board making process, bending test and tension strength test were conducted for the purpose of surveying the rupture stress of bio-boards.

The results indicate that under all experimental conditions, making boards using raw materials of corn straws was successful and the board making processes in this research were feasible. The rupture stress of bio-board A is in range of 6.23MPa ~ 10.84MPa and the rupture stress of bio-board B is in range of 12.90MPa ~ 16.95MPa. The average of rupture stress of bio-board B is 14.36MPa and 1.6 times greater compared with that of bio-board A 8.54MPa.

For chapter 2 indicated that the pressures in compressing process and the refining degree of a grinder in grinding process for board making might affect the strength of bio-board, As a result, the two important factors were investigated in Chapter 3 and chapter 4 respectively. Five stages pressures of 2MPa, 4MPa, 6MPa, 8MPa, 10MPa were applied to make boards. Besides, in chapter 5 bio-boards LFB (long fiber board) and SFB (short fiber board) with two different lengths of fibers were prepared. After bending and tension strength tests were performed, rupture stress, modulus of elasticity and static toughness data of bio-board were obtained. The rupture stress varied in the range of 21.25MPa ~ 30.78MPa in the bending test. On the other hand, the range of rupture stress of 4.49MPa ~ 15.15MPa appeared in the tensile strength tests. Rupture stress of bio-boards implied that rupture stress of five bio-boards were different, however, the strength of bio-board was affected slightly by pressures. The average range of Young's modulus of bio-board is 1.4GPa~1.8GPa. Static toughness is larger as pressure becomes higher. With 10MPa pressure bio-board has maximum static toughness 85J. For the investigation of refining degree, the results showed that the average of rupture stress varied in the range of 34.52MPa ~ 39.67MPa for LFB. On the other hand, rupture stress range of 37.9MPa ~ 41.25MPa appeared in SFB in bending test. In tensile strength, rupture stress varied in the range of 16.14MPa ~ 23.82MPa for LFB. On the other hand, rupture stress range of 20.69MPa ~ 27.41MPa appeared in SFB. The rupture stress of LFB and SFB resulted that generally rupture stress of SFB is greater than the one of LFB. Short fiber had more influence than longer fiber the strength of bio-board.

Finally, the comparison between corn straw bio-board and wheat straw in chapter 6 and the comparison with other chemical compound materials were studied and discussed. Strength test was considered as one of the main investigated methods to compare the mechanical properties of corn straw bio-board and other materials.

Basic mechanical properties of bio-board were investigated and the results proved that bio-board could be created for use a packaging material, for heat insulation in architecture, and as a mulch film for agricultural purposes.

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Chapter 1. Introduction

1.1 Background and significance of this study

Barthes (1957) called polyethylene plastics a miraculous substance, however, it has been regarded as the worst invention of 20th century by scientists (Dan Fletcher, 2010). Plastic brings human many conveniences and can be used in a near infinite number of ways (2013). In contrast, it threatens the environment as it is generally refined from fossil oil. According to an investigation from Biomass Energy Centre (2010), because of plastic's low cost, people use it without considering the effects. Efforts have been made to reduce its environmental impacts by shifting to the idea of sustainable development, but much more effort needs to be made (Jose, 2002).

Moreover, excessive consumption of fossil resources particularly in large urban areas has resulted high levels of pollution during the last few decades. To realize carbon dioxide reduction targets as specified in the Kyoto Protocol as well as to decrease reliance and dependence on the supply of fossil resources (Nibedita, 2012), countries across the world have considered and directed state policies towards the effective utilization of biomass for meeting their future energy demands. Therefore, research into a new substitute for fossil-derived materials is under consideration by scientists and other experts (Jorgelina, 2006).

The only other naturally-occurring, energy-containing carbon resource known that is large enough to be used as a substitute for fossil fuels is biomass. Biomass is all non-fossil organic materials that have intrinsic chemical energy content. In this study, environmental-friendly material is going to be developed using biomass resources. It means that many natural plant fibers can be universally utilized, then plant fibers return to organic manure after it is used to bury in soil. Biomass material utilization is significant for future life because when fossil resource is died out, many fossil products could continue alive in human life. Furthermore, processing biomass material as raw material with all the experimental processes does not bring any environmental burden, because one of chemical adhesive are used in the experimental process, which is special and unique characteristic from other similar biomass material researches (Itou, 2011). This research also investigates biomass material's mechanical properties in order to supply for different fields.

1.2 Definition of biomass material

Biomass is biological material derived from living, or recently living organisms. It most often refers to plants or plant-based materials, which are specifically called lignocellulosic biomass (2012). Biomass includes plant or animal matter that can be converted into fibers or other industrial chemicals, including biofuels. Industrial biomass can be grown from numerous types of plants, including miscanthus, switchgrass(Brechbill, 2008), hemp, corn, poplar, willow, sorghum, sugarcane, bamboo (Volk, 2000) and a variety of tree species, ranging from eucalyptus to oil palm (palm oil). In the context of biomass for energy this is often used to mean both plant and animal derived material. Within that definition from the Japanese Society of Plant Physiologists, there are five basic categories of material: agricultural residues, virgin wood, energy crops, food waste, and finally industrial waste and co-products such as broken up pallets and textiles.

Biomass resource can be utilized substantially in endless number of times, on the basic rail of carbon circulation by photosynthetic process. On the other hand, a fossil resource is limited to a transitory use in principle. Additionally the traversal emission of CO_2 caused by fossil combustion gives influence on global climate (Fig.1-1). R=resource (Yokoyama, 2008).

plant

[Biomass R.] → (use) → CO_2(・ ・ → Atmospheric CO_2)

[Fossil R.] → (use) → CO_2(・ ・ → Atmospheric CO_2) → CO_2 accumulation/air

Fig. 1-1 Comparison of biomass and fossil system on Carbon cycling

1.3 Utilization of biomass material in the world

The world's energy markets rely heavily on the fossil fuels coal, petroleum crude oil, and natural gas as sources of energy, fuels, and chemicals. Since millions of years are required to form fossil fuels in the earth, their reserves are finite and subject to depletion as they are consumed. The only other naturally-occurring, energy-containing carbon resource known that is large enough to be used as a substitute for fossil fuels is biomass. Biomass is all non-fossil organic materials that have intrinsic chemical energy content. Thus, as an important renewable raw material, biomass has become the focus of public attention.

Table 1-1 Biomass and other energy sources: production and consumption in the world

	1998 production quad btu	***percent Of total production***	***1998 consumption***
oil	152.0	40.0%	73.60 million barrels/day
Natural gas	85.5	22.5%	82.20 tcf/year
Coal	88.6	23.3%	5.01 billion tons/year
Nuclear	24.5	6.5%	2.30 trillion kWh/year
Hydroelectric	26.6	7.0%	2.60 trillion kWh/year
Biomass (other)	2.5	0.7%	196.00 billion kWh/year
Total	397.7	100%	

Source: EIA, 1998

World production of biomass is estimated at 146 billion metric tons a year, mostly wild plant growth (Cuff and Young, 1980). Biomass fuel is a renewable energy source and its importance will increase as national energy policy and strategy focuses more heavily on renewable sources and conservation. Biomass power plants have advantages over fossil-fuel plants because their pollution emissions are less. Biomass production and consumption in the world is given Table 1-1.(Mustafa Balat, 2005)

1.3.1 **Asian area**

Panasonic Malaysia developed an environmentally friendly material system for manufacturing kenaf (*Hibiscus cannabinus*) particle board. The process decreased pollution, thus conserving Malaysia&s rich coral reef ecosystem. The technique for manufacturing kenaf board was originally developed in cooperation with Kyoto University using kenaf grown in China. In 2005, Panasonic succeed in developing a process for growing kenaf in Malaysia that was suitable for manufacturing high quality kenaf particle board. This process produces 30% waste, but the fiber is burned to provide electric power for the manufacturing plant and the ash is returned to fields to fertilize kenaf. The process of kenaf board manufacture is shown in figure 1-2 (Fujiwara, 2010).

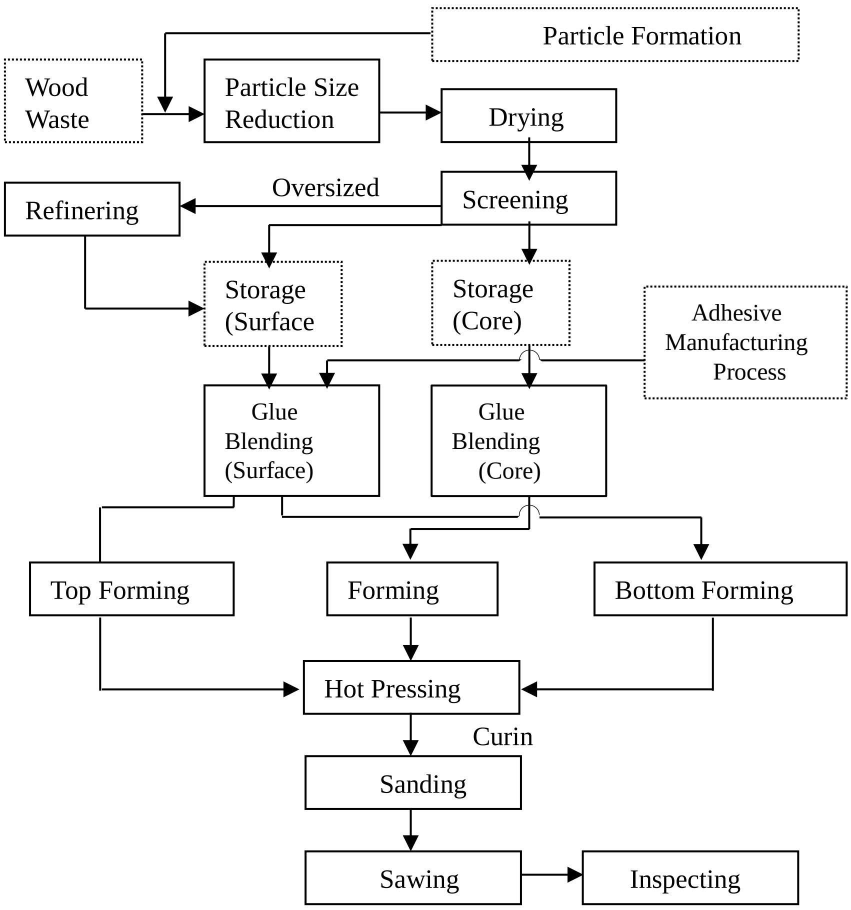


Fig. 1-2 Kenaf board Manufacturing Process.

1.3.2 **The American continent**

Forest by-products, such as wood residues, are common in the United States (Shahab, 2001). The forest products industry in North American traditionally uses sawmill residues and small round logs as raw materials to manufacture fiberboard. However, growing concern about the environment has led to changes of forest management practices, resulting in significant reduction in wood harvest from our national forests in the midst of growing demands. Increasing import of timber and fiber supply is only a temporary solution. That is why it is a clear potential for the use of agricultural fiber in manufacturing what have traditionally been wood based products (Bowyer,1995; Clancy-Hepturn, 1998).

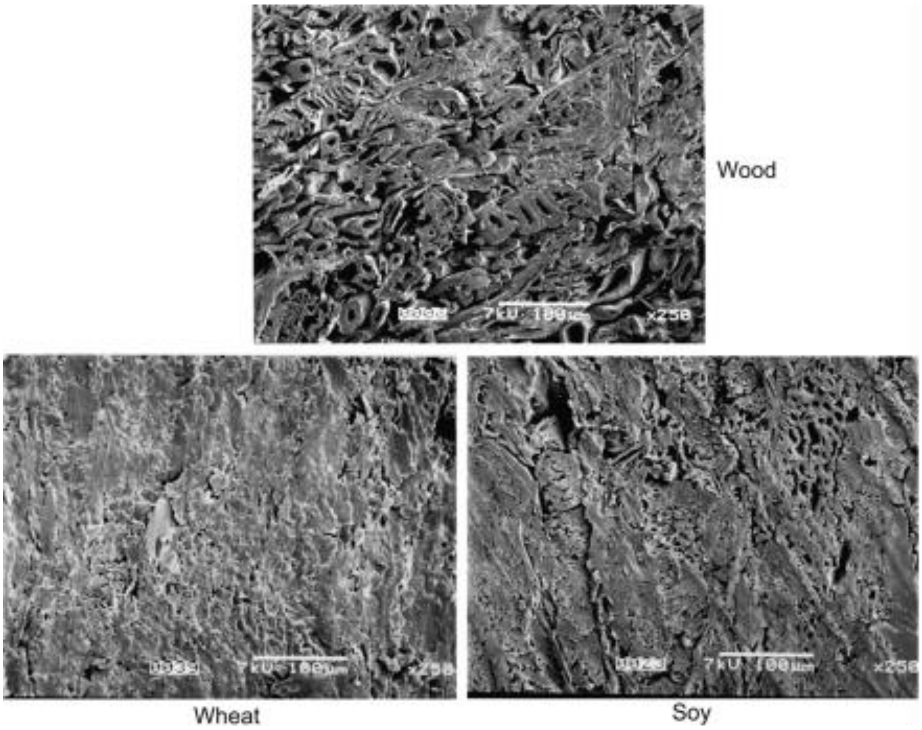


Fig. 1-3 Scanning electron micrographs of cut surfaces of MDF made from the three fiber types.

It has been estimated that 400 million dry tons of crop residues are annually produced in the United States (DOE, 2003). Since 1995, there has been a proliferation of new manufacturing facilities in Canada and US to produce composite panels from agricultural residues.Most of these manufacturing plants produce particleboard from wheat straw.

In 2007, X. Philip Ye (2007) and his partners had studied a medium density fiberboards made from wheat and soybean straw. The MDF properties modulus of elasticity, modulus of rupture, internal bond strength, thickness swell, and screw holding capacity were evaluated. The Scanning electron micrographs of cut surfaces of MDF made from the three fiber types were shown in figure 1-3.

1.4 World utilization of corn and corn straw

Maize as a kind of herbaceous biomass is used all over the world. Conversion forms include physical conversion thermochemical conversion.

1.4.1 Structure and physiology

The maize plant is often 2.5 m (meters) (8 ft) in height, though some natural strains can grow 12 m (40 ft) (Karl 2013). The stem has the appearance of a bamboo cane and is commonly composed of 20 internodes of 18 cm (7 in) length. (Stevenson, 1972) (Wellhausen, 1952) A leaf grows from each node, which is generally 9 cm (3.5 in) in width and 120 cm (4 ft) in length, as fig.1-4 shows structure of maize, Ears develop above a few of the leaves in the midsection of the plant, between the stem and leaf sheath, elongating by 3 mm/day. They are female inflorescences, tightly enveloped by several layers of ear leaves commonly called husks. Certain varieties of maize have been bred to produce many additional developed ears. These are the source of the "baby corn" used as a vegetable in Asian cuisine.

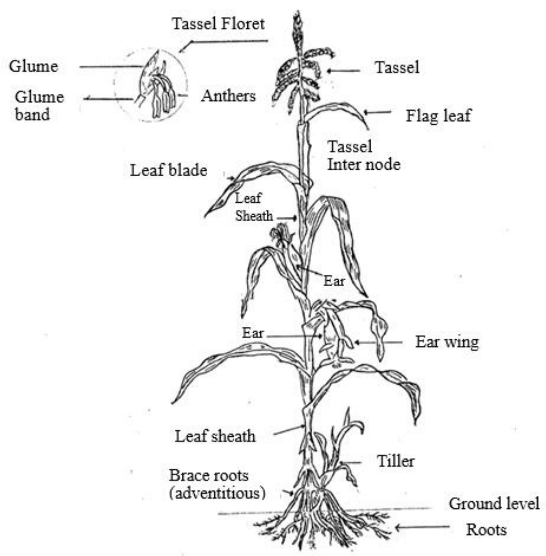


Fig. 1-4 Structure of maize

The apex of the stem ends in the tassel, an inflorescence of male flowers. When the tassel is mature and conditions are suitably warm and dry, anthers on the tassel dehisce and release pollen. Maize pollen is anemophilous (dispersed by wind), and because of its large settling velocity, most pollen falls within a few meters of the tassel.

Elongated stigmas, called silks, emerge from the whorl of husk leaves at the end of the ear. They are often pale yellow and 7 in (178 mm) in length, like tufts of hair in appearance. At the end of each is a carpel, which may develop into a "kernel" if fertilized by a pollen grain. The pericarp of the fruit is fused with the seed coat referred to as "caryopsis", typical of the grasses, and the entire kernel is often referred to as the "seed". The cob is close to a multiple fruit in structure, except that the individual fruits (the kernels) never fuse into a single mass. The grains are about the size of peas, and adhere in regular rows around a white, pithy substance, which forms the ear (maximum size of kernel in subspecies is reputedly 2.5 cm/1 in (Grobman, Alexander (1961).). An ear commonly holds 600 kernels. Young ears can be consumed raw, with the cob and silk, but as the plant matures (usually during the summer months), the cob becomes tougher and the silk dries to inedibility. By the end of the growing season, the kernels dry out and become difficult to chew without cooking them tender first in boiling water.

1.4.2 **Production situation of corn in the world**

Even though corn or maize is not one of the most economic crops in Thailand, there has been an increasing utilization of corn cob in Power generation and solid fuel in order to achieve zero-waste philosophy in corn processing industry.

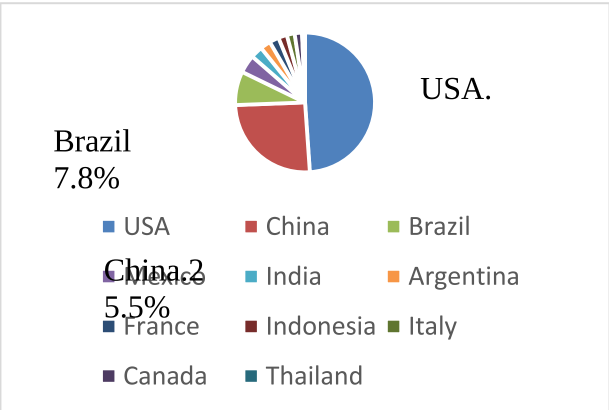


Fig. 1-5 World leading countries in maize production in 2006.

The figure 1-5 and figure 1-6 above shows the 2006 production of maize around the world, where USA is accounted for almost half of the world production and also with the highest yield in the world. Although China has a similar amount of land for maize plantation to USA, the lower yield in China makes its production only half of the USA figure.

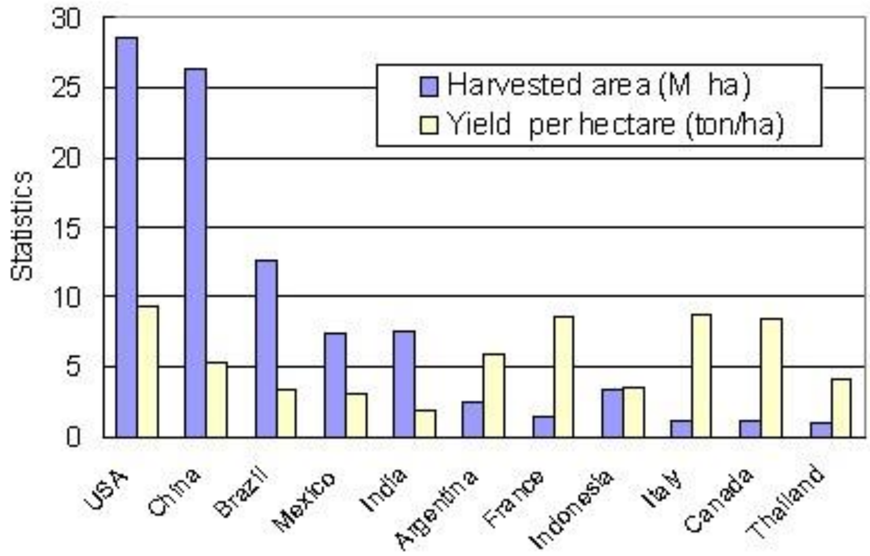


Fig. 1-6 World harvested area and yield of maize in 2006

1.4.3 **Composition of lignocellulosic biomass**

Lignocellulosic biomass is composed primarily of carbohydrate polymers (cellulose and hemicellulose) and phenolic polymers (lignin). Lower concentrations of various other compounds, such as proteins, acids, salts, and minerals, are also present.

(a) Cellulose

(30–50% of total feedstock dry matter) is a glucose polymer linked by ß–1,4 glycosidic bonds. The basic building block of this linear polymer is cellubiose, a glucose-glucose dimer (dimer: two simpler molecules—monomers—combined to form a polymer). Hydrolysis of cellulose results in individual glucose monomers. This process is also known as saccharification.

(b) Hemicellulose

(20–40% of total feedstock dry matter) is a short, highly branched polymer of five-carbon (C5) and six-carbon (C6) sugars. Specifically, hemicellulose contains xylose and arabinose (C5 sugars) and galactose, glucose, and mannose (C6 sugars).

Hemicellulose is more readily hydrolyzed compared to cellulose because of its branched, amorphous nature. A major product of hemicellulose hydrolysis is the C5 sugar xylose. Figure 1-7-c shows the structural formula of xylan. Other hemicelluloses include glucomannan, but all hemicelluloses vary in amounts depending on tree species and the part of the plant.

(b) Lignin

(15–25% of total feedstock dry matter), a polyphenolic structural constituent of plants, is the largest non-carbohydrate fraction of lignocellulose. Unlike cellulose and hemicellulose, lignin cannot be utilized in fermentation processes.

It is a compound whose constituent units, phenylpropane and its derivatives, are bonded 3-dimensionally. Its structure is complex and not yet fully understood. Figure 1-7-d shows a constituent unit. Its complex 3-dimensional structure is decomposed with difficulty by microorganisms and chemicals, and its function is therefore thought to be conferring mechanical strength and protection. Cellulose, hemicellulose, and lignin are universally found in many kinds of biomass, which are the most plentiful natural carbon resources on the earth.

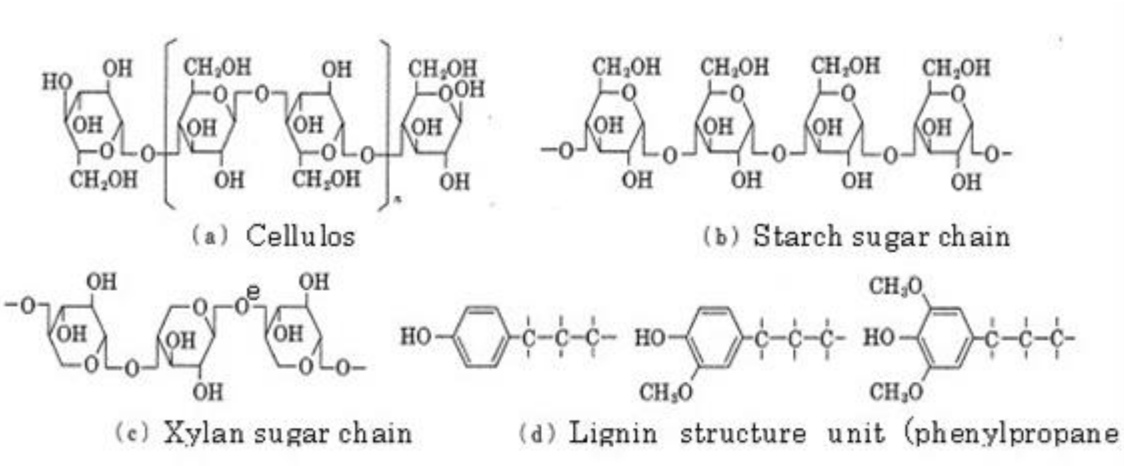


Fig. 1-7 Chemical structures of major biomass components

Agriculture-derived biomass, specifically crop residues from corn and small grains and dedicated perennial grasses such as switchgrass, are emphasized in this report. Table 1-2 lists general characteristics of these and other potential biomass resources (Pordesimo, 2005).

Table 1-2 Composition of potential lignocellulosic biomass resources

Unit: **% of dry matter**

	cellulose	**Hemi -cellulose**	**lignin**	**Acid detergent lignin**	**Crude protein**	**Ash**
Crop residues						
Corn stover	38	26	19	4	5	6
Wheat straw	38	29	15	9	4	6
Soybean	33	14	-	14	5	6
Rye straw	31	25	-	3	3	6
Barley straw	42	28	-	7	7	11

In corn, soluble solids rapidly decrease and lignin and xylan increase shortly after grain physiological maturity Table 1-3 (Pordesimo, L.O. 2005)

Table 1-3 Changes in composition of corn straw and leaf with crop maturity

Unit: **% of dry matter**

Corn stalk	**Late dent (110d*)**	**Physiological maturity(153d)**	**Post physiological maturity (220d)**
Structural glucan	35	35	35
Xylan	16	22	23
Lignin	15	20	19
Protein	3	4	4
Soluble solids	15	4	4
Corn leaf	18	23	32
Structural glucan	2	17	22
Xylan	4	13	16
Lignin	8	8	4
Protein	35	8	6

Source: Pordesimo, L.O. 2005

*Days after planting

1.5 Conventional paper pulp processing

The modem manufacture of paper evolved from an ancient art first developed in China, ca. 105 A.D. Although the modem product differs considerably from its ancestral materials, papermaking retains distinct similarities to the processes developed by Ts'ai Lun in the Imperial Chinese Court. ' In principle, paper is made by: 1) pulping, to separate and clean the fibers; 2) beating and refining the fibers; 3) diluting. to form a thin fiber slurry, suspended in solution; 4) forming a web of fibers on a thin screen; 5) pressing the web to increase the density of the material; 6) drying to remove the remaining moisture; and 7) finishing, to provide a suitable surface for the intended end use.

All integrated pulp and paper mills involve the same general steps in the manufacture of pulp and paper. These steps include: 1) raw material preparation (e.g., debarking and chipping); 2) me- chanical and/or chemical separation of the wood fibers [i.e., grinding, refining, or digestion (cook- ing)] to dissolve the lignin and extractives; 3) removal of coloring agents (primarily residual lig- nin) by bleaching; and 4) paper formation and manufacture.

In this research, bio-board making processes are similar to that of papermaking process, because agricultural fiber is similar to wood fiber in composition and function. Cellulose and hemicellulose existing in agri-fibers plays an important role in fiber-to-fiber bonding in board making process. This point become a significance guiding principle

1.6 Purpose and content of this study

The main purpose of the present study deals with the manufacturing process for a green biomass board using corn straw (stem and leaves). The unique different process from other fiberboard is that any addition of binder was used in fiber bonding process. Hydrogen bonding connection is considered as the basic board making principle. The factor of pressures, length of fibers, pressing temperature in forming process on the strength of biomass board are investigated and discussed.

Mechanical properties such as density, bending strength, bending rupture stress, tensile strength, tensile rupture stress, modulus of elasticity, wet basic moisture content in bio-board are also studied after board making experiment.

Chapter 2. Bio-board Making Process

2.1 Introduction

Although traditional methods are available for corn straw utilization such as animal feed, fuel for cooking, and house-heating. Presently, corn straw as one of the biomass resources is utilized in bioenergy field universally. However, for rural families more than 50 percent of corn straw remains and most of them are still burnt in the field. This kind of disposal method has caused environment concerns as it lead to air pollution and has a bad influence on taking off and landing of airplane. Therefore, effective technologies for corn straw disposal and utilization need to be developed.

In general, this research consists of two parts that develop processes for making a bio-board using corn straw and some standard strength tests applied to investigate the material properties in next part. Processes for making board is still under studied by applying different experimental conditions. In strength test, materials properties including tensile strength and tensile strength rupture stress are investigated.

2.2 Basic principle for fiber bonding

Corn stover-to-grain ratios are about 1:1 on a dry matter basis, and corn stover is about 38% cellulose, 26% hemicellulose, and 19% lignin. Wheat straw yields, dry matter basis, are about 1.3–1.4 lb straw per 1 lb grain. Wheat straw is about 38% cellulose, 29% hemicellulose, and 15% lignin. Cellulose is a polymeric sugar (polysaccharide) made up of repeating 1,4-ß-an hydroglucose units connected to each other by 8-ether linkages. The long 1inear chains of cellulose permit the hydroxyl functional groups on each anhydroglucose unit to interact with hydroxyl groups on adjacent chains through hydrogen bonding and van der Waals forces according to DoKyoung Lee (2007).

The basic principle of board producing experiment in this study is that take advantage of the hydrogen bonding between cellulose chains and hydrogen molecules showed in Figure 2.2.1. Cellulose is hydrophilic and insoluble in water with the properties of highly resilient and impact resistant. Likewise, Hemicellulose can be used in material for its function of resistant deforming and highly adhesive. To strengthen the material, it dehydrates through pressurization and heating treatment of cellulose and re-establishment connection between caudal ends of cellulose chain. The major components of lignocellulosic wastes are cellulose, hemicellulose and lignin in varying quantities. Therefore main components help producing bio-board effectively by using a physical hot-pressing method.

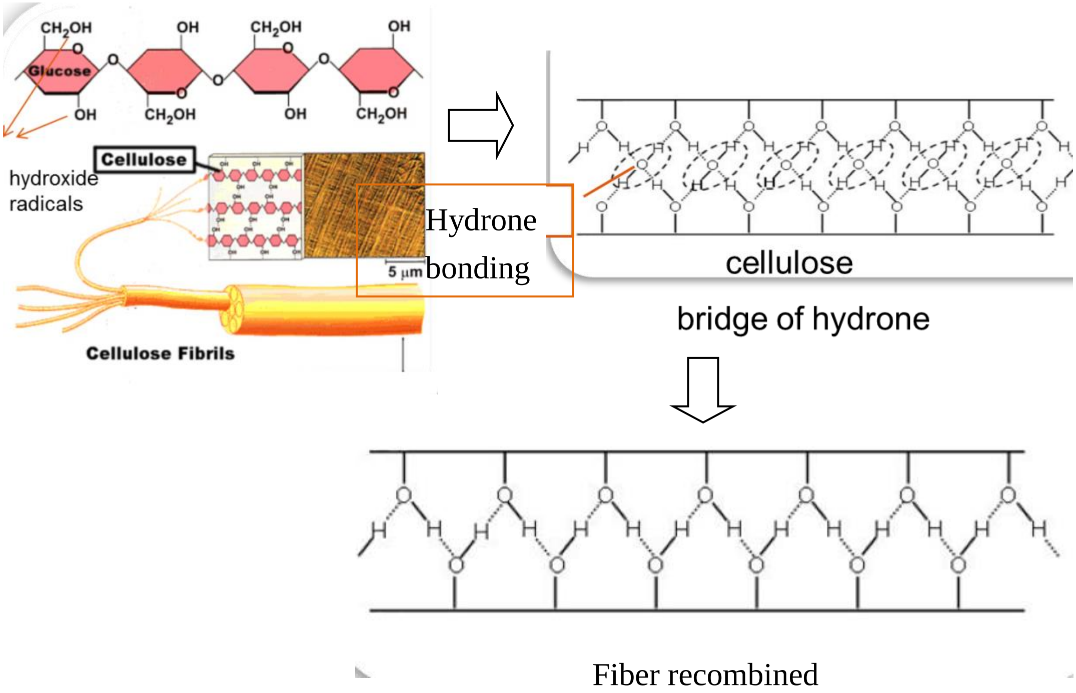


Fig. 2-1 Process of hydrogen bonding between cellulose chains

2.3 Preparation for experimental material

Normal corn cultivar were collected from the farm in Mie University and died in a well-ventilated building at room temperature for about 1 month (Richey, 1982). Corn straw (cookies and leaves) was only selected for board making Conditions

For pretreatment process, long corn straws were cut into small chips when they were in a dry state and ground by an electric mixer for about 5minutes. Straw fibers would be separated in accordance with the length of corn fiber is about 0.5-1.5mm (Yang, 2001). Second, water was added to the refined corn straw and they were soaked in a calorstat for 72h at 35℃;

Table 2-1 presents the different conditions for producing bio-board in this study. Pretreatment temperature is 70℃ using a calorstat and an ambient temperature of 20℃ was used during the experiment. The hot-presser can supply maximum pressure of 11MPa, and therefore, a distinct difference is designed to determine between compression pressures and drying pressures using 4MPa and 7MPa for each board production.

Table 2-1 Board production conditions

Board number	Soaking temperature [℃]	compressing pressure [MPa]	Drying pressure [MPa]	Pressing Temperature[℃]	Drying Temperature[℃]
No.A	70	4	4	100	110
No.B	20	7	7	100	110

2.4 Board making process

Figure 2-2 shows the flowchart of process for bio-board production. After maceration they were transferred to an electric stone mill where they were ground with water-filled. During this process, more active hydroxy groups of cellulose chain would be exposed and as corn straw were de-fibrated in which micromolecule were made into microfibers for a foundation of physical adsorption. It was good for separated fibers to recombine with each other in compression process. The cubic compression mold was made with specific dimensions of 100mm x 100mm x 25mm and some accessory include a metal block, a steel plate and two meshes in the compression process. For the purpose of evaporation where there is an array of 7x7 mm punched holes with 2mm in diameter in cubic mold, metal block and the plate.

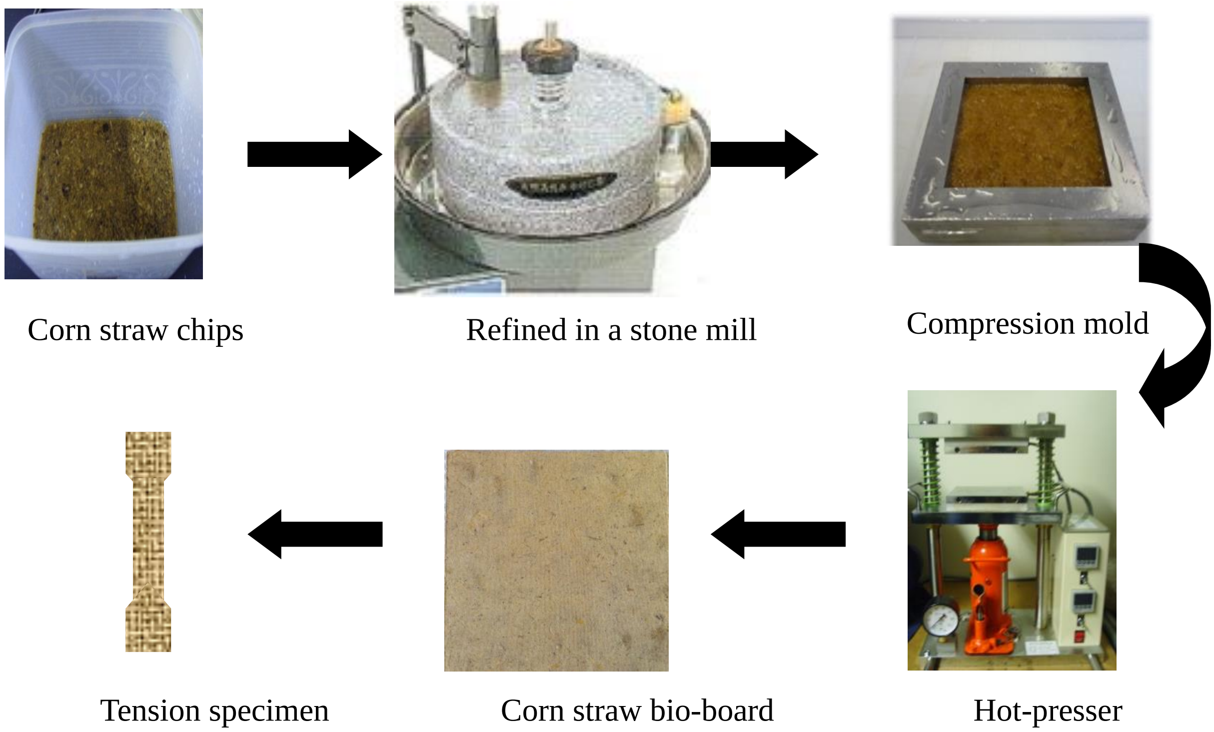


Fig. 2-2 Flowchart of process for bio-board production

2.5 Strength test

Specimens for tensile test were taken from Board A, Board B and polyethylene plastic tray using for food container. Each specimen was cut to an equal length-to-width ratio (5:1) to set in the cradle of the tension apparatus with special end grips owing to the delicate nature of biomass material and the smooth surface. The grips consisted of inner sand paper that attached to the surface of the specimen, providing extra frictional force as the jaws were tightened. Each specimen was clamped within the jaws of the load frame and pulled at a uniform rate of 10mm/sec. Separate sensor was fixed to the motor which provided power to the extensometer that was used to measure accurately the strain resulting from the tension loading. Figure 2-3 is a picture showing the tension test setup.

Data were logged by an Amplifier and A/D convert. Calculations for the rupture stress required measuring the physical parameters of the specimen (i.e., cross-sectional area). Because it was a kind of cellulosic material, the surface of specimen is not as well as metal. The thickness and width were determined 5 times finally using the average value to calculate cross-sectional area. The rupture stress is given by Eq. 2.1:

$$\sigma = \frac{P}{A} \quad (2.1)$$

in which, σ is the normal stress; P is the tensile load and A is the cross-sectional area.

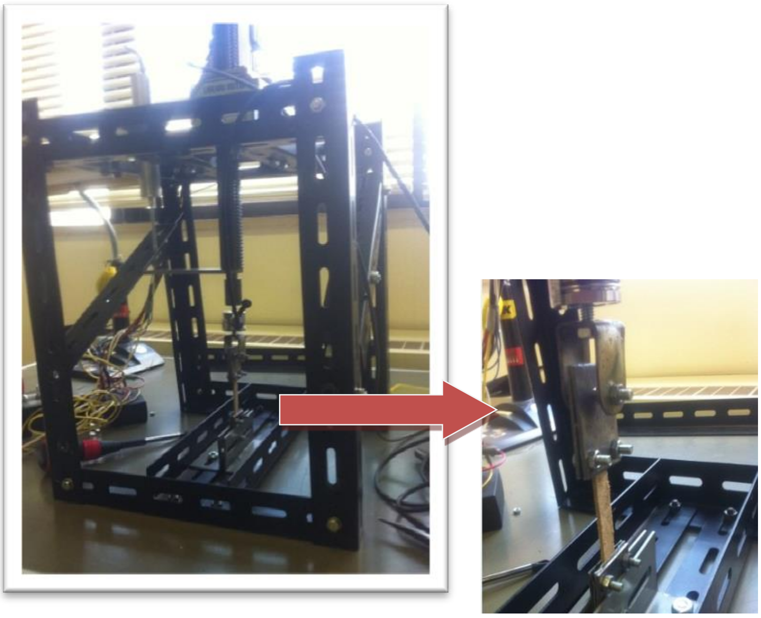


Fig. 2-3 Tensile test setup for bio-board.

2.6 Measurement of moisture content

The moisture content of biomass board in our experiment is an important physical parameter of bio-based materials. In this study the moisture content of each specimen was measured after the tensile test subsequently. The tested pieces were dried in a an oven set at 105℃ for 24h. The moisture content measurement is in accordance with wet base and dry base given by Eq.2.2 and Eq.2.3,

$$MC(W.B) = \frac{M_b - M_a}{M_a} \quad (2.2)$$

$$MC(D.B) = \frac{M_b - M_a}{M_a} \quad (2.3)$$

In which M_a is the masses of specimen after drying (g), M_b is the natural masses of specimen before drying (g). *W.B* is moisture content measured under a wet benchmark and *D.B* is moisture content measured under a dry benchmark. In order to measure the moisture content of pieces which were in a tension state, finished specimen was put in a hermetic aluminium vessel immediately after taken from the cradle of tension apparatus.

2.7 Results and discussions

2.7.1 **Board making**

Bio-board was produced with different soaking temperatures, compressing pressures, drying pressures after refining, de-fibrating, compressing and drying processes. As figure 2-4 shows it is successful to make Board A and Board B using the experimental conditions and processes developed in this research. Some distributed fibers could be seen obviously on the surface of bio-board. However it was smooth as the metal mashed was used to prevent from being stick to the mold. After measured the volume size and checked the weight, the specific gravities were calculated for 0.857g/cm^3 ~1.014 g/cm^3. Thus, the average could be 0.929 g/cm^3.

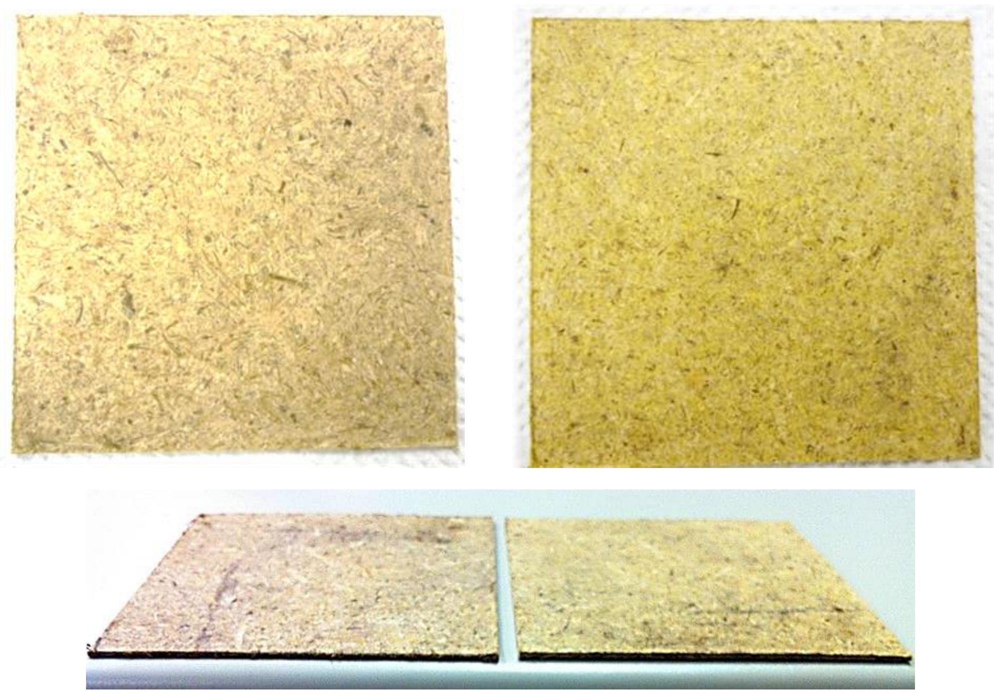


Fig. 2-4 Appearance of bio-board A and B 1(left: board A, right: board B, bottom: section part)

2.7.2 **Strength test**

Specimens before strength test were shown in figure 2-5 They were numbered and displayed for the dimension of specimen was measured. After tensile strength test, specimens failed. The photo in figure 2-6 shows the broken specimens image. It can be seen from the broken specimen that fracture point of each specimen appeared in different position of specimen. In addition, corn straw fiber which is clearly observed on the edge of fracture point.

Fig. 2-5 Specimens before tensile strength test

Fig. 2-6 Specimens after tensile strength test

Figure 2-7 shows the tensile stress-strain curve of six specimens cut from board A. In the strength test, bio-board specimen is pulled and deformed, stress increased with the increase of strain for all the specimens. This process refers to be similar with elastic deformation of metal. All of specimens show similar behavior. When the specimen is fractured, the stress reached the maximum value then decreased sharply, in the end stress varied to zero.

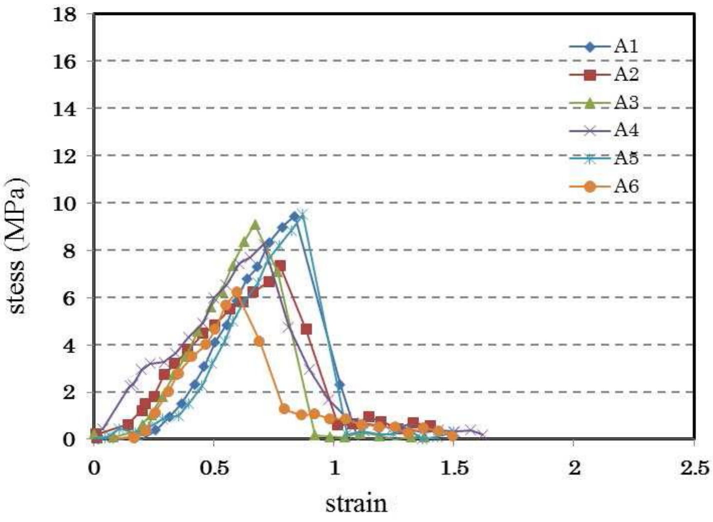


Fig. 2-7 Tensile stress-strain curve of board A

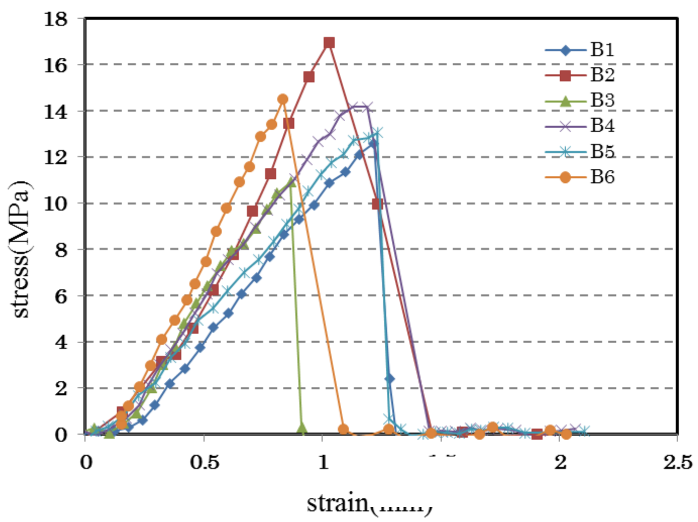


Fig. 2-8 Tensile stress-strain curve of board B

The stress-strain curve of each specimen has unique characteristics and shows a different maximum stress. These changes are considered that arrangement of natural fibers and the density of corn straw are different. Recombination of corn straw fibers is also different at different areas in one bio-board.

In the case of the other condition, bio-board was produced and the stress-strain curve is shown in figure 2-8. As well as bio-board A, the stress of 6 specimens increased with the increase of strain. However, the inclination of stress-strain curve for each specimen shows slight difference. When the specimen is fractured, the stress reached the maximum value then decreased sharply, in the end stress varied to zero. The stress-strain curve of each specimen shows a different maximum stress. The difference of maximum stress for each specimen is attributed to the combination of natural fibers and the density of corn straw at different areas in one bio-board.

Rupture stress of bio-board B is greater than that of bio-board A. The maximum pressure in the forming process of bio-board B was 7MPa, it is higher than 4MPa applied in bio-board A. This result indicated that maximum pressure applied in the forming process effects the stress of bio-board significantly. Therefore, the pressure applied on bio-board in forming process is important for the stress of bio-board produced.

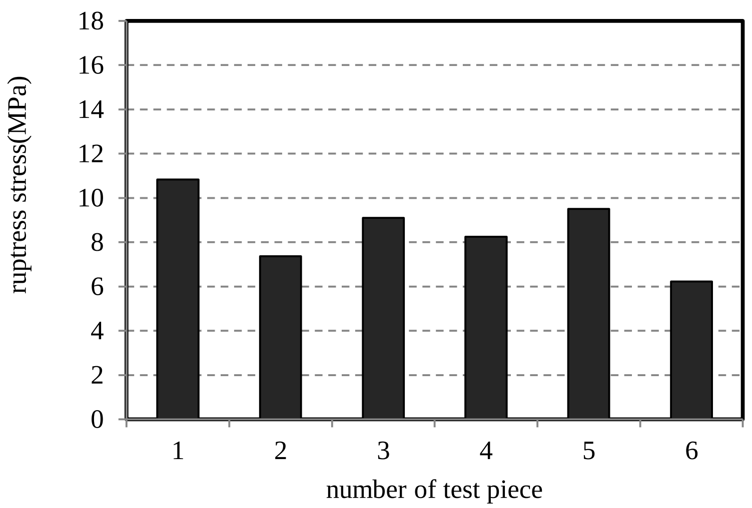


Fig. 2-9 Tensile rupture stress of bio-board A

Tensile rupture stress of each specimen cut from Bio-board A is presented in figure 2-9. The rupture stress of 6 specimens are different, minimum rupture stress is 6.23MPa and maximum rupture stress is 10.84MPa. Rupture stress varied in the range of 6.23MPa ~ 10.84MPa. The average of rupture stress for 6 specimens is 8.54MPa.

The 6 specimens were produced under the same producing condition, because they were cut off from bio-board A. However, the rupture stress of 6 specimens shows different values. The reason can be considered that the combination of natural fibers and the density of corn straw at different areas in one Bio-board are different.

Figure 2-10 shows the rupture stress of bio-board B. The 6 specimens cut off from bio-board B but their rupture stresses are different. The minimum rupture stress is 12.90MPa and maximum rupture stress is 16.95MPa. The rupture stress varied in the range of 12.90MPa ~ 16.95MPa. The average of rupture stress for 6 specimens is 14.36MPa. As the same as bio-board A, the difference of rupture stress for6 specimens was affected by the different combination of natural fibers and the density of corn straw at different areas in one Bio-board.

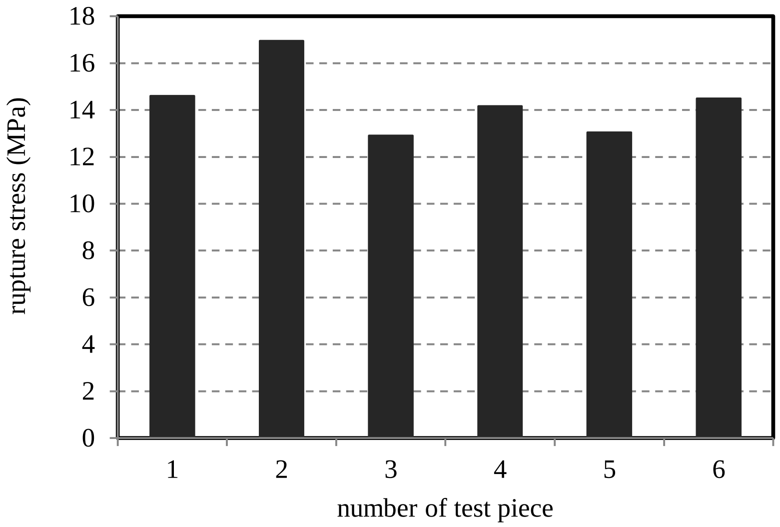


Fig. 2-10 Tensile rupture stress of bio-board B

From the strength test result of two Bio-boards, the rupture stress of bio-board B is greater than that of bio-board A. The average of rupture stress of bio-board B with 14.36MPa is 1.6 times greater compared with that of bio-board A. bio-board B was produced under a condition of compressing and drying pressure 7MPa, which is higher than 4MPa applied in bio-board A production. It can be indicated that pressure of compressing and drying process influence the strength of bio-board directly. As bio-board is a kind of biologic materials, by their very nature, are complex composite structures whose components are intimately connected. Thus, pressure is a stimulus to connection of corn straw fibers.

In case of polyethylene plastic using for food container, was tested by tensile test. The rupture stress of polyethylene plastic is 0.72MPa. It can be indicated that strengths of bio-board were 8~23 times greater as compared with the polystyrene plastic. Bio-board produced in this experiment can be applied to food container in the way of strength.

2.7.3 **Moisture content**

Results of moisture contents are illustrated in figure 2-9 which is moisture content of wet base and in figure 2-10 which shows moisture content of dry base of bio-boards. It can be observed from the results in figure 2-11 that moisture content of bio-board A which was made by 4MPa varied slightly among the six specimens. The lowest value is 2% which is from number 1 specimen and the highest value 7% came from number 4 specimen. On the other hand, moisture content of bio-board B reveals that great variation happened in different specimen. The value ranges in 2%~9% which comes from bio-board B made by 7MPa.

In addition, moisture content of bio-board A and B in dry base was shown in figure 2-12 The trend varies in moisture content is similar to that in wet base. It ranges in 2%~8% of bio-board A meanwhile the moisture content of bio-board B is in the range of 3%~10%.

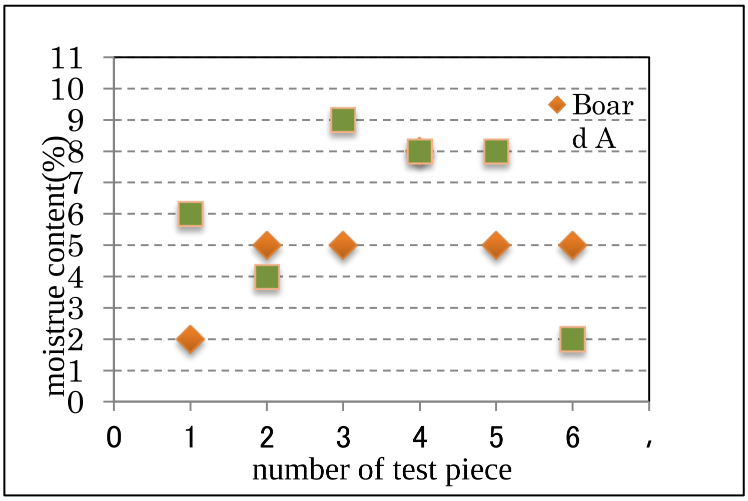


Fig. 2-11 Moisture contents of bio-board A and B in wet base.

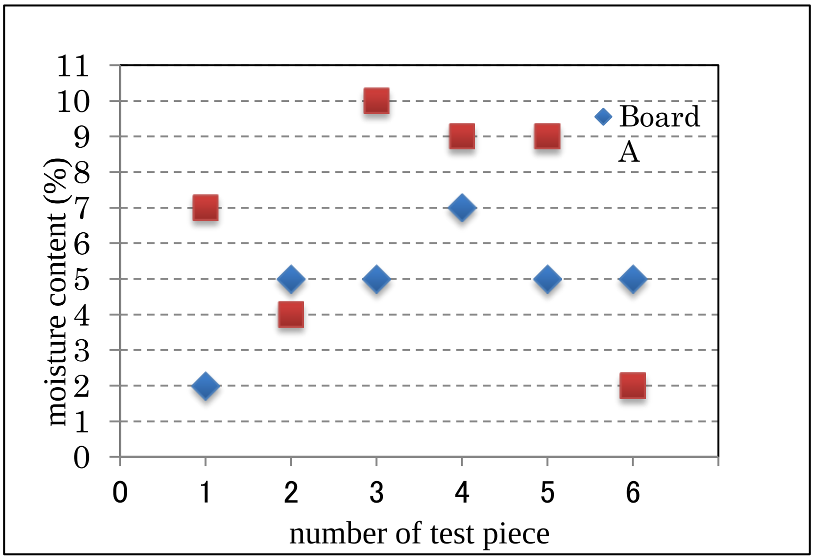


Fig. 2-12 Moisture contents of bio-board A and B in day base.

2.8 Conclusions

The main objective of this study is to investigate the possibility of producing biodegradable biomass materials using corn straw. The processes of refining, defibrating, forming and drying were applied to produce Bio-board with the experimental conditions of compressing pressure 4MPa and 7MPa, drying temperature 110 degrees centigrade. Two pieces of Bio-board were produced and their properties were investigated by tensile test.

(1) Bio-board can be produced under experimental conditions. The experimental result indicates that all the processes applied in this experiment are suitable to produce Bio-board and the experimental conditions were considered to be appropriate.

(2) The rupture stress of bio-board A is in range of 6.23MPa ~ 10.84MPa and the rupture stress of bio-board B is in range of 12.90MPa ~ 16.95MPa. The rupture stress of one bio-board are totally different, because of the differences in combination of natural fibers in forming process, and the density of corn straw at different areas in one Bio-board.

(3) The average of rupture stress of Bio-board B is 14.36MPa and 1.6 times greater

compared with that of Bio-board A 8.54MPa. Therefore, compressing pressure is an important factor for the strength of Bio-board.

(4) The tensile test result of polystyrene plastic used in food container shows that the rupture stress is 0.72MPa. The rupture stress of Bio-board is 8.6~23.5 times greater in comparison to the polystyrene plastic.

Chapter 3. Effect of pressures on the strength of bio-board

3.1 Introduction

According to previous research about making bio-board using corn straw, the results shows us that bio-board can be made successfully using corn straw and all the process applied in the experiment is feasible.

In this chapter, the present research is dealing with proved manufacturing process for bio-board using corn straw (stem and leaves). The principle of board making which is different from other fiberboard is still hydrogen bonding. Effect of the pressure applied in forming process on the strength of biomass board is investigated. Additionally, the other mechanical properties such as rupture stress, Young's modulus and strain energy will also be studied and discussed.

3.2 Materials and methods

3.2.1 **Board making experiments**

(1) Basic principle

As Martin A. Hubbe (2006) says cellulose and hemicellulose, two of the main components of fibers, are covered with hydroxyl groups. The oxygen atoms in these groups are able to hydrogen bond to hydrogen atoms on adjacent fibers or water molecules. Drying of bio-board causes some fiber-to-fiber hydrogen bonds to take the place of fiber-to-water hydrogen bonds.

(2) Board making process

Sweet corn (Zea mays L. convar. mays) straws were used in this work. Sowing date was on May 1st and harvested on August 10th (Liu 2009) at the Mie University Bio-resource Department's experimental farm. Figure 3-1 shows maize farm. After harvesting, grains were removed. Stem and leaves were left in a ventilated storage air-dried for two months.

Fig. 3-1 present situation of sweet corn cultivation in this research

The five processes applied in previous experiment are used in the present study which are cutting, soaking, grinding, compressing and drying showed in figure 3-2. Compressing and drying procedures are carried out together and called “the forming process”.

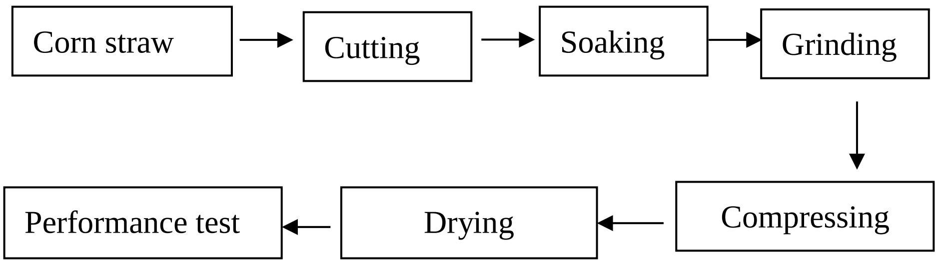


Fig. 3-2 Flow chart of Bio-board making process

During pretreatment, dry corn straws were cut into chips using an electric cutter, then soaked in water at 22℃ for 168 hours for softening the straw fiber. In soaking process, corn straw fiber bundles absorbed moisture from water condition. It is easier to soften fiber bundles in wet condition than destroy the structure of lignocellulose fibers in a dry state. Soaking process is a preparation to the fiberization of corn straws.

Soaked straw was then fiberized (pulped) by using an atmospheric refiner with conical blades in figure 3-3 (Model A Beatfiner. Satomi. Corp.). The motor capacity is 11kw×4p-200, 60Hz, rotational speed is 1750 r.p.m (60Hz). The maximum flux control is 0.05-0.1m3/min. Air pressure is 0.6MPa required. Grinding part is an assembling conical cutters with blades. Dimension of cutter is 2.5mm×3.0mm×8°(blade width×slot width×blade angle).

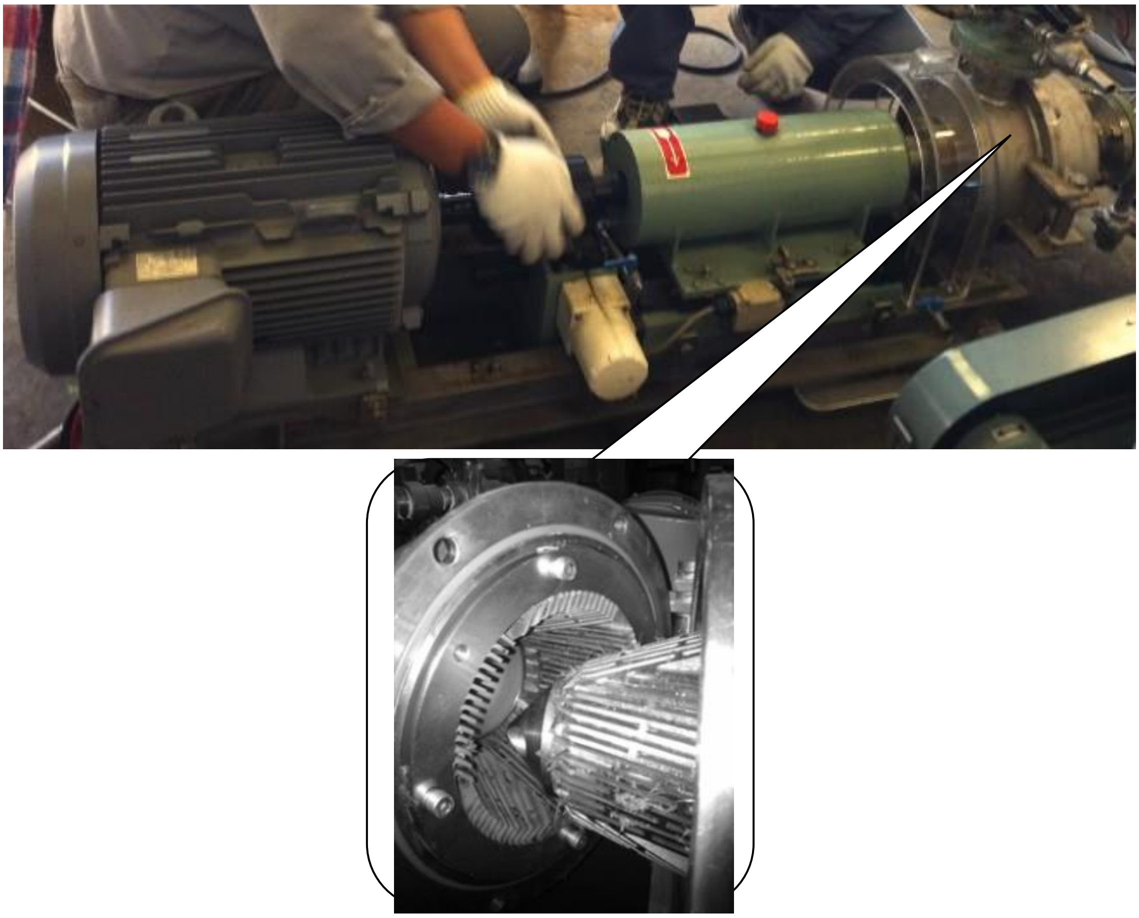


Fig. 3-3 An atmospheric refiner and the detail blades

Fiberization of corn straws at atmospheric pressure was carried out by passing the damp cut straw along with running water through the refiner' rotating blades. During grinding process, fiber bundles would be fiberized by milling. Accordingly, milled-corn straw was sieved to possess particle size using a screen with 2mm×2mm hole size. The ground straws were fractionated into a fine fraction which possessed particle size of 0.5mm~2mm. Grinding process was done with water. Therefore, corn straw pulp as figure 2 shows was prepared before compressing.

A closed stainless steel die with some accessories including metal block, meshes, were designed which enabled obtaining one square board, 100 mm long ,100 mm width and 40mm depth. The calculated amount of ground corn straw was carefully filled in the die, and prepressed by for pressing excess water out from the die. Holes were drilled in the bottom of die, metal block and plate 2 mm in diameter, in a 7 mm×7 mm grid allowing water to escape in the forming process.

The desired pressure was applied at the maximum temperature of 110 ºC. It took 8-10 minutes until the die containing the samples reach the maximum temperature. Forming experimental conditions are displayed in Table 3-1. As Pan (2009) described during forming process hydrogen bonds hold the chains firmly together side-by-side and forming micro fibrils with high tensile strength and water inside of bio-board could be also evaporated by high temperature and pressure.

Table 3-1 Condition of Bio-board production

bio-board No.	**pressure (MPa)**	**dying temp. (℃)**
A1,A2	2	110
B1,B2	4	110
C1,C2	6	110
D1,D2	8	110
E1,E2	10	110

3.2.2 **Strength tests**

Bending tests and tensile strength tests were conducted for the purpose of analyzing the mechanical properties of bio-board.

In the Three-Point Bending Test shown in figure 3-4, five bio-boards named A1 to E1 were trimmed to 50mm × 10mm × 1.2mm. 25 rectangular beam specimens following standard JIS procedures and recommendations (JIS Z2248:1996) were prepared. All dimensions were measured with an accuracy of + 0.02 mm. Capacity of 100 N load cell was fixed on a motor, applied at a uniform rate of 0.57 mm/s in its vertical direction. Furthermore, the deformation signal was measured by a potentiometer. Both signals of force and deformation were transmitted into an amplifier and A/D convertor then logged in a computer. The bending stress of bio-board was obtained by the quotients of bending moment and section modulus of the specimen. Rupture stress was defined by quotients of maximum bending moment and section modulus of specimen when the specimen was fractured. The classic formula (William, 1957) for determining the bending stress is:

$$\sigma_b = \frac{3PL_s}{2ba^2} \qquad (3.1)$$

Where P = force at fracture of test specimen; *Ls* = bearing distance between supports; b = width of test specimen; a = thickness of test specimen.

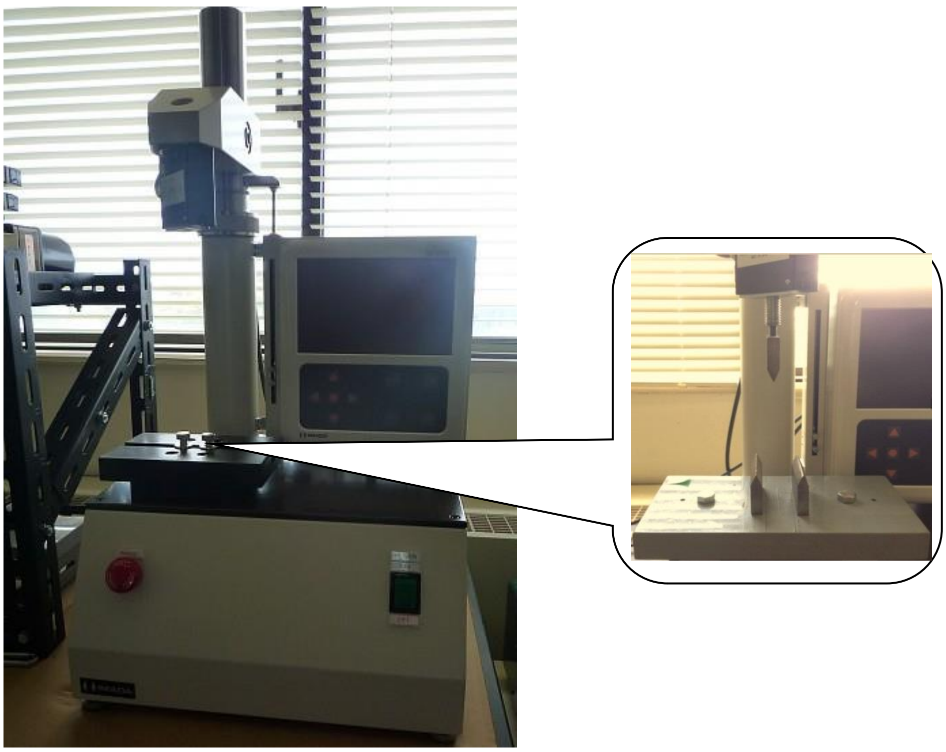


Fig. 3-4 Three-point-bending test machine

To determine the internal bond strength, tensile strength tests were done under deformation control by using a universal testing machine shown in figure 3-5. Speed of cross-head was 15mm/min. 20 specimens taken from five bio-boards named A2 ~ E2 were subject to tensile strength tests following standard JIS procedures and recommendations (JIS z 2201) illustrated in figure 3-6.

Normal stress is defined by the quotients of axial load applied on specimen and original cross-sectional area of the specimen. Rupture stress was obtained when the axial load reaches to maximum value while the specimen was fractured. Rupture stress is expressed below:

$$\sigma_t = \frac{P}{A} \tag{3.2}$$

Where P = the maximum axial load, A= original cross-sectional area of gauge section.

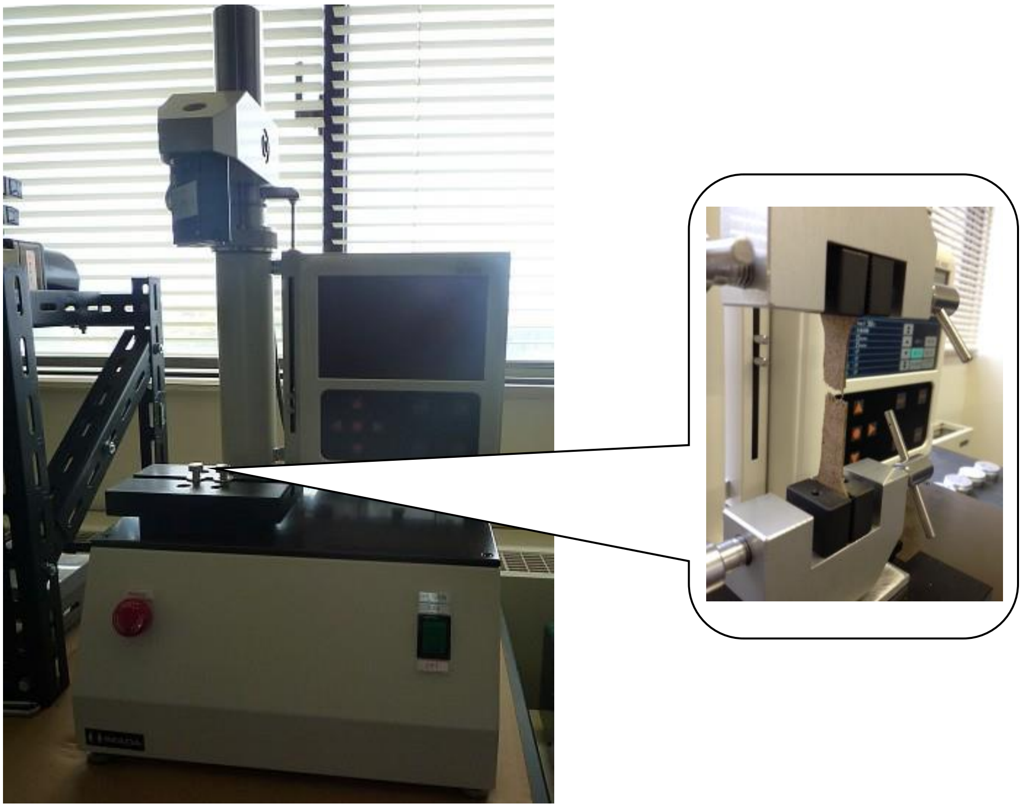
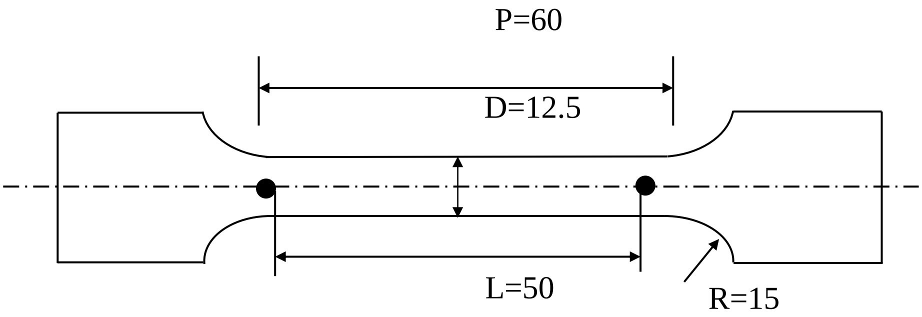


Fig. 3-5 Universal testing machine for tensile test



P: length of reduced section, L: Gage length
R: Radius of fillet, mm. D: Diameter

Fig. 3-6 Dimension of specimen for tensile strength tests

3.2.3 **Measurement of density and moisture content**

Ten bio-boards were made to demonstrate the five experimental conditions. For each of the experimental conditions, two bio-boards were made. They are named A1, A2, ~, E1, E2.

Thickness of bio-board is measured as below. On one board three horizontal lines and three perpendicular lines were drawn then the area of bio-board was divided into sixteen square blocks. The area of each block was 25 mm×25mm. For thickness measurement, eight points at the outer side of four blocks in the center of board were chosen. Thus, densities of bio-board were determined as follows:

Density = hot presser dry weight (g)/ sample volume (cm3)

Moisture content analyze were done after strength tests: Numbered specimens were cut into chips and weighted, oven dried at 100℃ till constant weight, and moisture percentage was calculated according to JSPP (2007).

3.2.4 **Young's modulus and strain energy**

In tensile strength test, Young's modulus of bio-board was calculated from stress-strain curve. The definition of material toughness is the amount of energy per unit volume that a material can absorb before rupturing. It is also defined as a material's resistance to fracture when stressed. On the other hand, the strain energy pre unit volume is called the strain energy density and is the area underneath the stress-strain curve up to the point of deformation. According to definitions, static toughness of five bio-boards was calculated with formula (3.3).

$$U = \int_0^{\varepsilon_f} \sigma d\varepsilon \qquad (3.3)$$

Where, U= static toughness, σ= stress, ε= strain, ε_f = strain at fracture point.

3.3 Results and discussion

3.3.1 **Board making**

Bio-board of A1, B1, C1, D1, E1 were subjected to the bending test and A2, B2, C2, D2, E2 were used for the tensile strength test. The dimension of all bio-boards were 100 mm×100 mm in area and in the range of 1.27 mm-1.56 mm in thickness. Figure 3-7 shows the front and side of one bio-board. This experiment proved that Bio-board can be made successfully under all experimental conditions. Not only the Bio-board, but the processes for making the Bio-board were also shown to be successful.

Density of bio-board displayed in figure 3-8 indicates that bio-board has respective density at the range of 0.87g/cm3.-1.02g/cm3. With the five levels of pressure 2MPa to 10Mpa resulted in slight increase in the board density. Under condition of 2Mpa, board density is minimal. The maximum density was observed in the condition of 8Mpa.

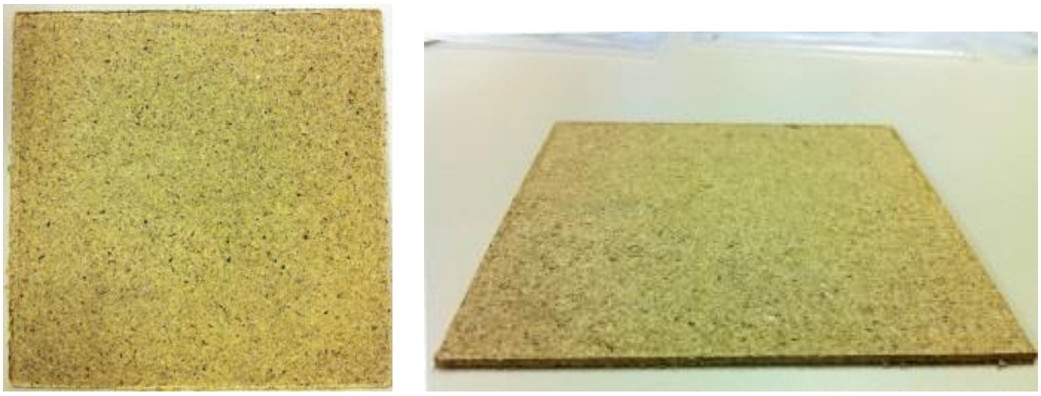


Fig. 3-7 Appearance of bio-board

The moisture contents of bio-board made in this study showed a range of 3% ~ 6% in wet base. Properties of density and wet-basis moisture content of bio-boards are similar to MDF (medium density fiberboard) 5 Type ~ 30 Type based on JIS A 5905-2003.

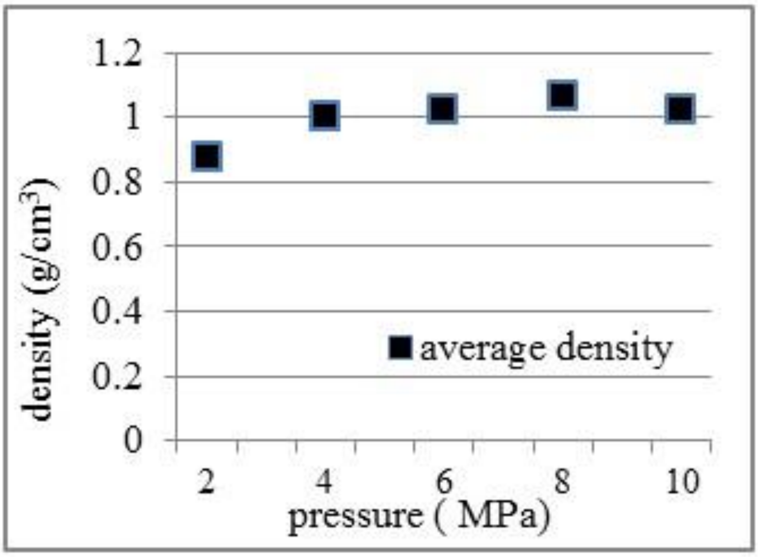
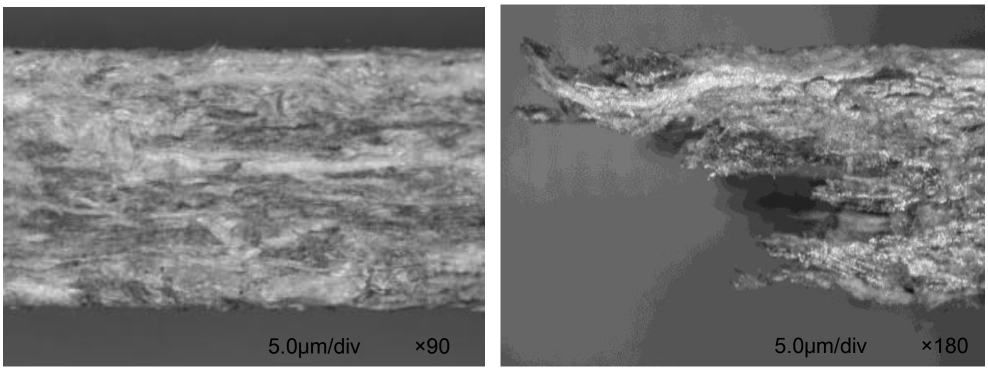


Fig. 3-8 Density of bio-boards

3.3.2 **Section of bio-board**

In figure 3-9 on the right shows the section image of specimen under electron microscope at magnifications. Corn straw fibers inside bio-board are observed compacted and displayed irregular. The image on the left shows a dentation fracture image of specimen. The fracture in specimen is rough. Uniform fibrous fracture is visible in the fragment. One reason must be considered that a bio-board is a composite. Its performance depends on the strength of its constituent units as well as their geometries and unit-to-unit bonding.



Cross section (right) , dentation fracture of bio-board (left)

Fig. 3-9 An electron micrograph of bio-board.

3.3.3 **Bending strength test**

The 25 specimens obtained from board A1 to board E1 were provided for bending test. The stress-deflection curves of five boards are shown in figure 3-10 to figure 3-14 changes of bending stress applied on five specimens are compared in the stress-deflection curves. Generally, stress value increased with the increase of deflection until reaching a maximum value. In addition, the curves of stress-deflection almost approach a straight line before the fractures occurred. Specimens fractured at approximately 2mm of deflection. Bending stress is called rupture stress when the specimen fractured. After specimen fractured, bending stress decreased sharply and is near to zero, however, bending stress still appears a few because some fibers were still remained connecting.

Compared to the other four specimens in one bio-board a distinctly highest peak value was observed in board A1-P1, B1-P1 and C1-P4. The stress-deflection curve describes unique characteristics and shows different modulus of elastic and rupture strength of each specimen.

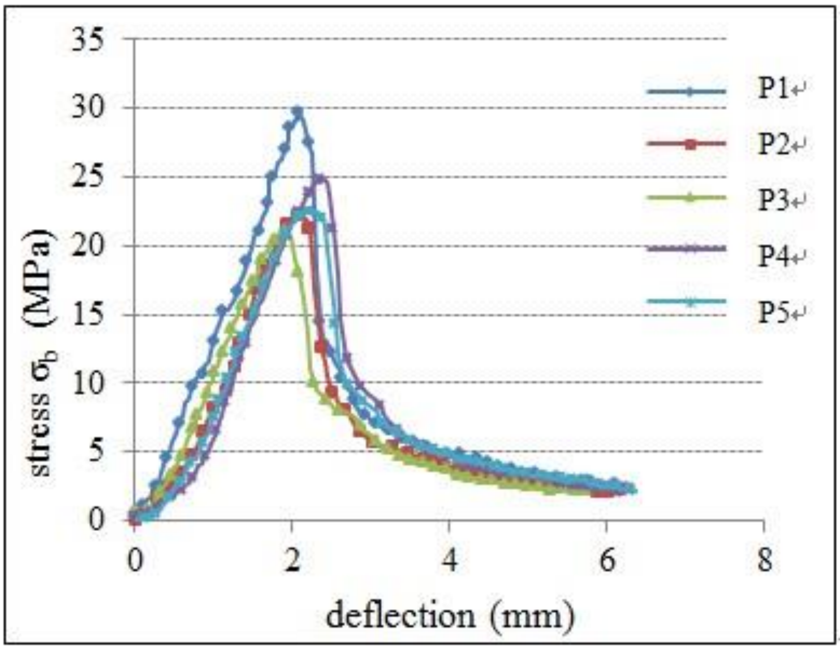


Fig. 3-10 Stress of bio-board A1

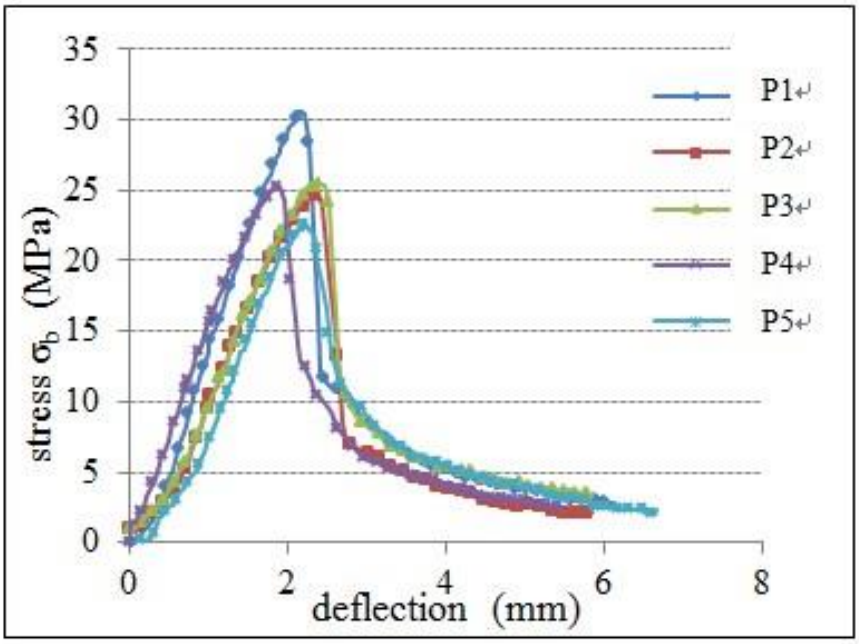


Fig. 3-11 Stress of bio-board B1

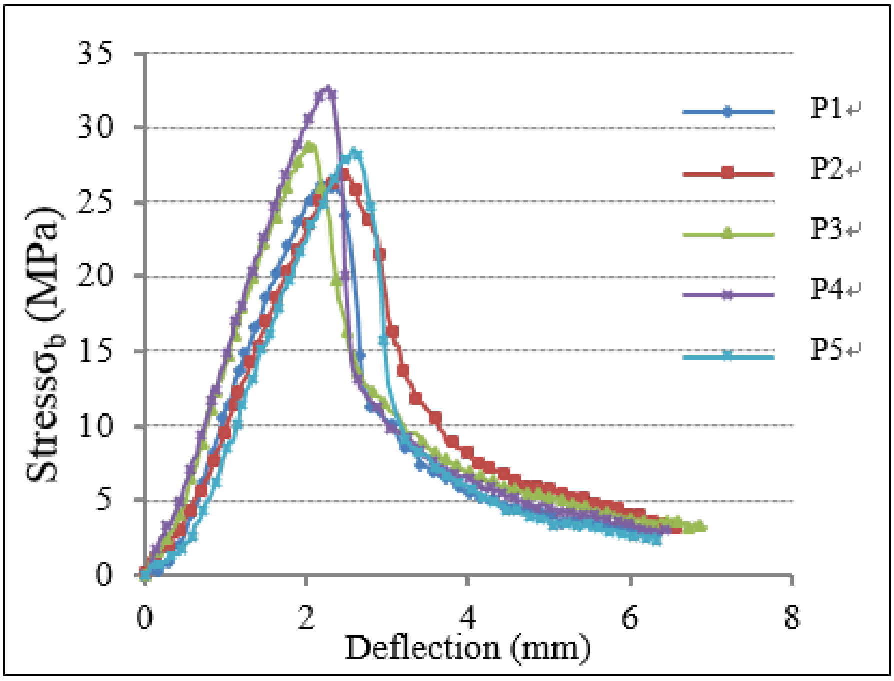


Fig. 3-12 Stress of bio-board C1

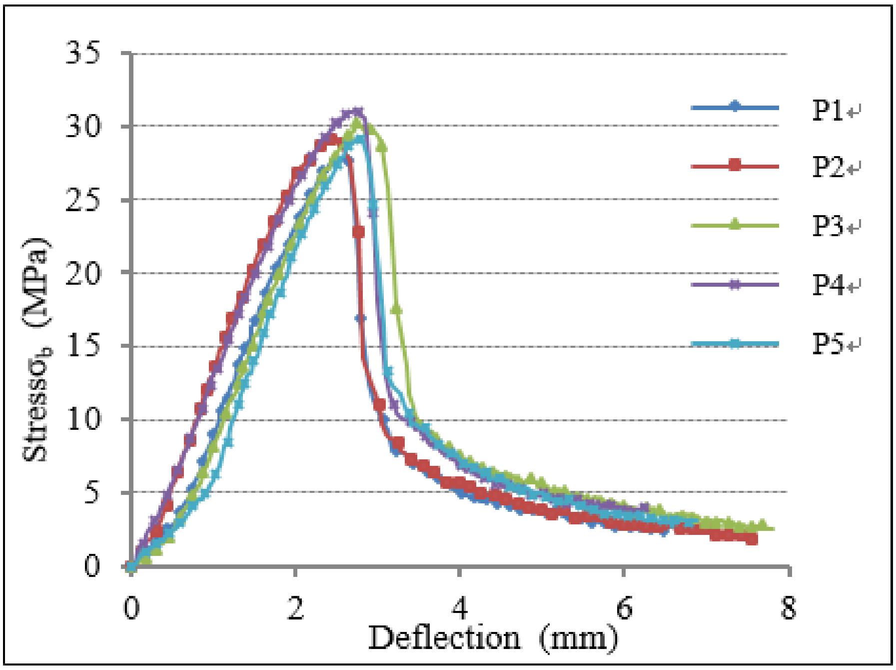


Fig. 3-13 Stress of bio-board D1

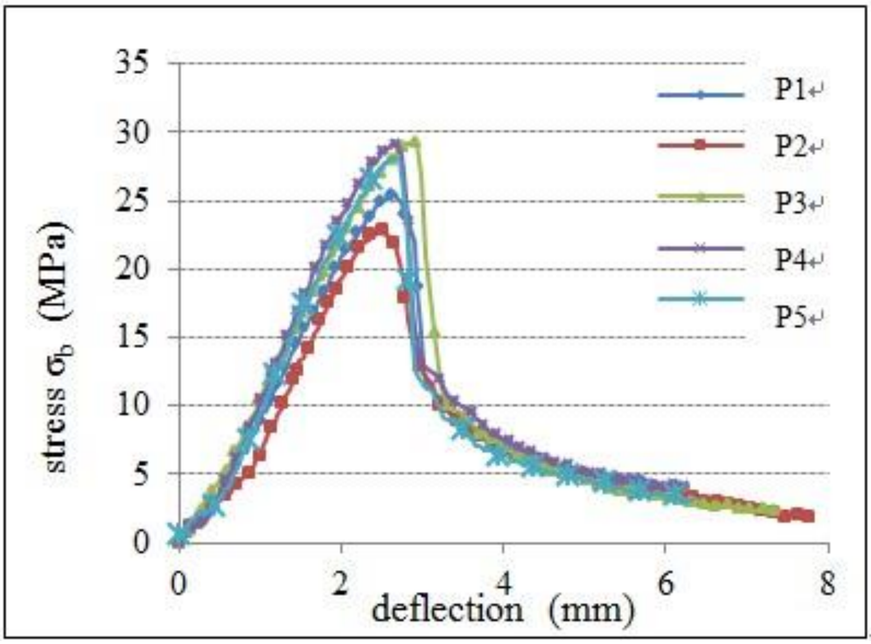


Fig. 3-14 Stress of bio-board E1

In figure 3-15, different rupture stress values bio-boards maximum value, average value and minimum value were calculated from stress-deflection curves. The results indicate that the higher pressure applied, the higher rupture stress was obtained, however, under the pressure of 10MPa rupture stress decreased slightly. The variability in density of bio-board also implies that at the pressure of 10Mpa density of board E1is lower than board D1. On the other hand, minimal variability in rupture stress was obtained in board D1.

The bending rupture stresses of board A1 to E1 is presented in figure3-15. According to the results of strength test, it implies that rupture stress of five bio-boards is obviously different. Maximum bending rupture stress 29.37Mpa occurred with the condition of 8Mpa. Therefore, results of bending strength test prove 8Mpa pressure applied in forming process is optimum condition to make bio-board.

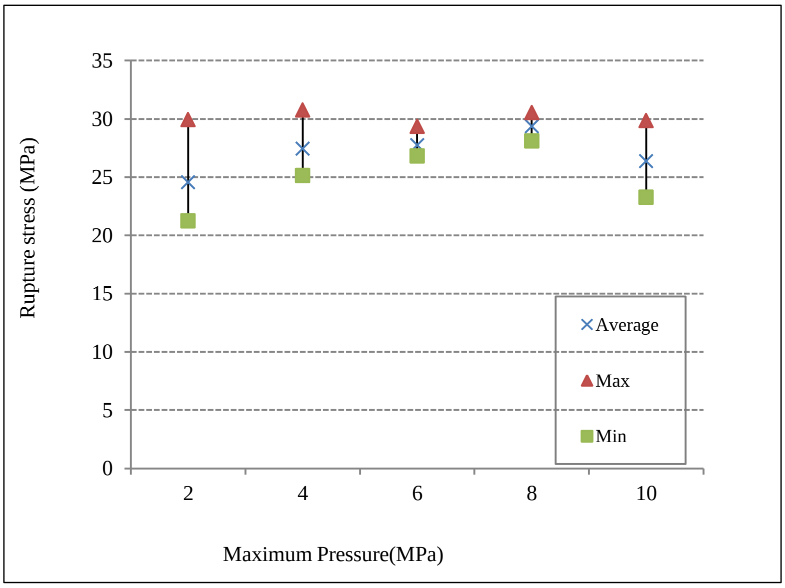


Fig. 3-15 Rupture stress of five provided Bio-board for bending test.

3.3.4 **Tensile strength test**

As an important mechanic property for bio-board study, tensile strength test was conducted. Boards named A2, B2, C2, D2, E2 were used for tensile strength tests and the stress-strain curves are shown in figure 3-16 to figure 3-20 All the curves indicates in the beginning of the stress-strain curve, tensile stress increases with the increase of strain, after the tensile stress reaches the maximum value, specimens were broken, then, tensile stress decreases suddenly to zero. The maximum value is called tensile rupture stress.

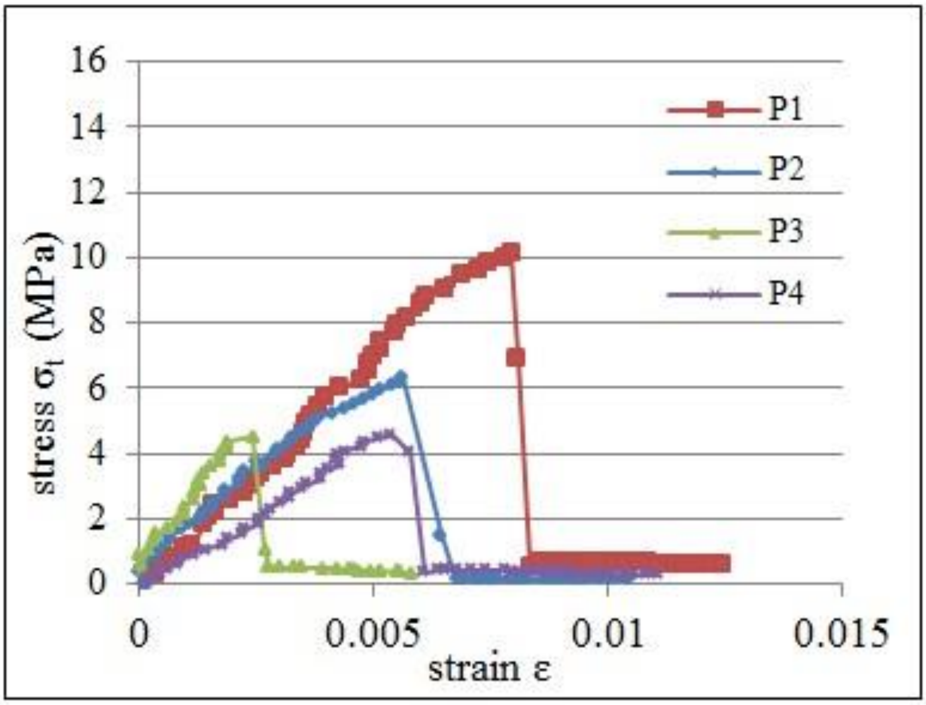


Fig. 3-16 Stress-strain curve of bio-board A2

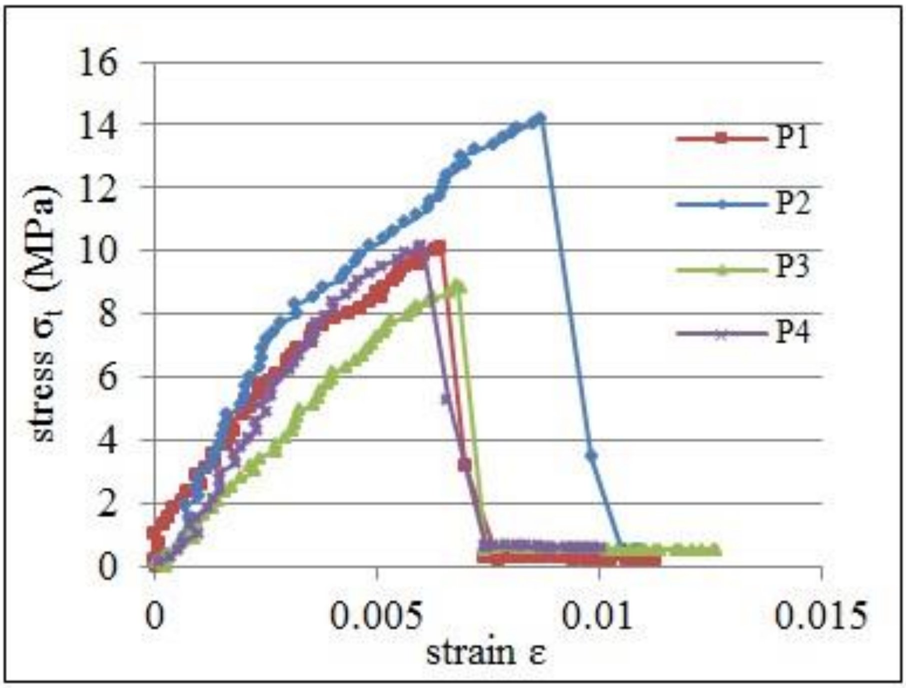


Fig. 3-17 Stress-strain curve of bio-board B2

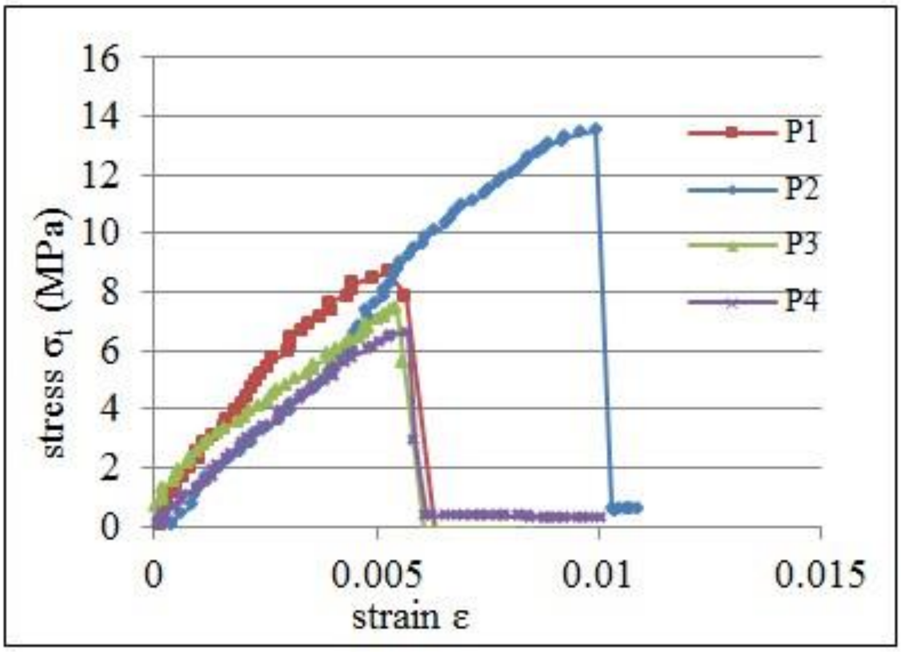


Fig. 3-18 Stress-strain curve of bio-board C2

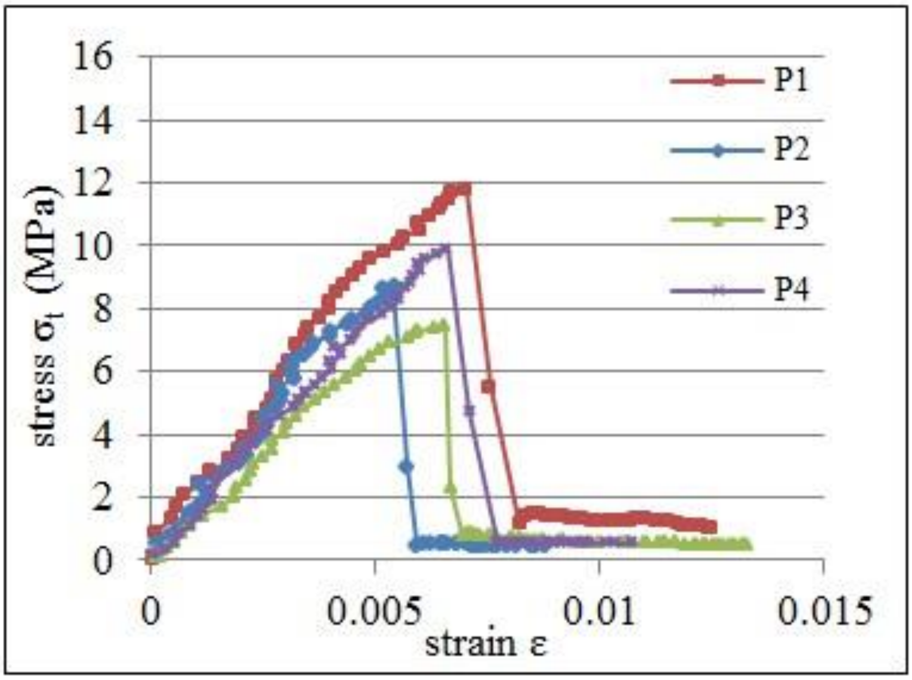


Fig. 3-19 Stress-strain curve of bio-board D2

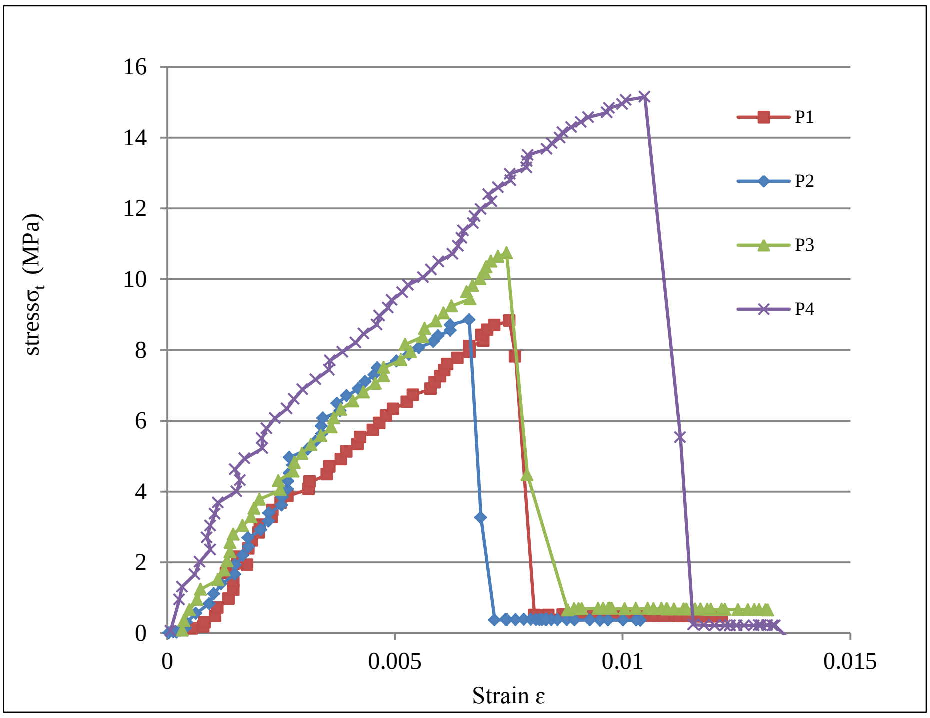


Fig. 3-20 Stress-strain curve of bio-board E2

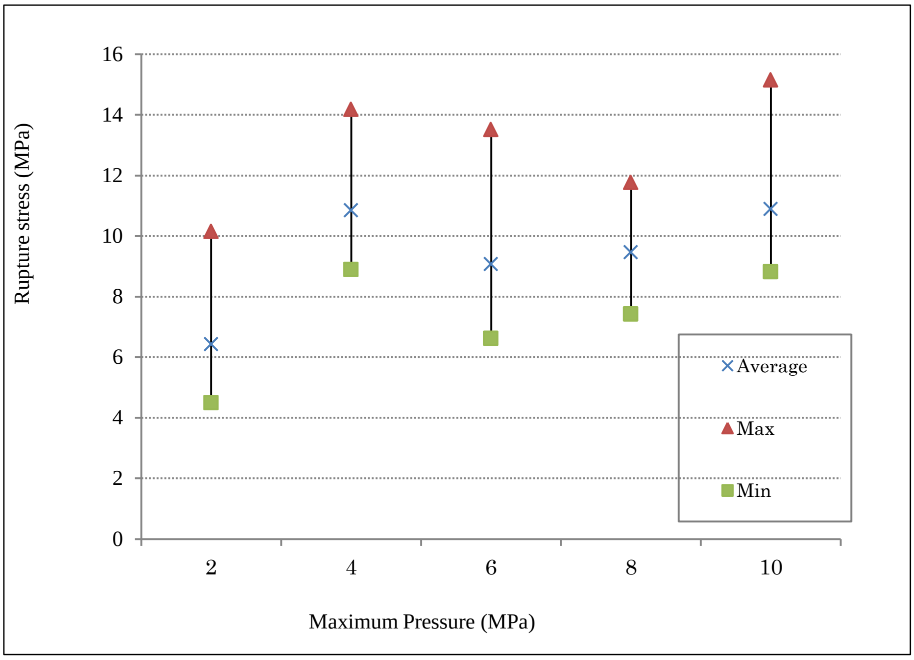


Fig. 3-21 Rupture stress of five provided Bio-board for tensile test

The tensile rupture stress of five bio-boards is shown in figure 3-21 with maximum value, average value and minimum value. Rupture stress in tension obtained from board A2 ~E2 showed a large variety between maximum stress and minimal stress in one bio-board. In table 3-2 rupture stress reveals that the maximum 10.89MPa was obtained in board E2 which was made by 10MPa pressure.

Compared to figure 12 showing bending rupture stress, rupture stress σ_{tf} is lower than bending rupture stress σ_{bf}. According to “An Introduction to the Building Standard Law NO.1452”, the bending stress of coniferous wood is higher than tensile stress because bio-board as well as wood is essentially composed of cellulose hemicelluloses, lignin, and extractives (Sjostrom 1993).

Bending rupture stress σ_{bf} and tensile strength rupture stress σ_{tf} are shown in table 3-2. This distinct variation could be explained by two reasons. First, Kageyama (1998) in his teaching materials stated that stress distribution of fiber material are more complex than metal. Stress distribution specimen in bending test performs in tension under neutral axis while above neutral axis specimen was suffered from compression behavior. For tensile strength test, specimen was suffered from tension force and only tensile stress distribution was in the area of thrust surface. Second, stress of fibrous bio-board may relate to fiber recombination because cellulose fiber has its own mechanical properties proved by Isogai (The society of Polymer Science, Japen. 2009).

Table 3-2 Average rupture stress of bio-board

	2MPa	4MPa	6MPa	8MPa	10MPa
σ_{bf} (MPa)	24.57	27.45	27.74	29.37	26.39
σ_{tf} (MPa)	6.43	10.85	9.07	9.46	10.89

3.3.1 **Young’s modulus and strain energy**

In tensile strength test, it seems that stress-strain curves almost approach a line before the specimens fracture. Therefore, the correlation between stress and strain on the approximate portion of stress-strain curve was modeled by a linear form. Young’s modulus of bio-boards were calculated and displayed in table 3-3. It has been found that the range of E in every bio-board is different. Four specimens of one board showed various deformation performances. The average range of Young's modulus of bio-board is 1.4GPa~1.8GPa.

Table 3-3 Young's modulus of bio-boards

pressure(MPa)	range of *E* (GPa)	average *E*(GPa)
2	0.8~2.2	1.4
4	1.4~1.9	1.8
6	1.3~1.9	1.6
8	1.3~1.6	1.6
10	1.2~1.7	1.5

Average static toughness of five bio-boards is displayed in table 3-4. The results found that pressure applied in forming process has a great influence on the specimens. Board E2 made with the pressure of 10MPa has the maximum static toughness 85KPa, in the opposite, Minimum static toughness 48.3KPa was obtained in board A2 which was made with the pressure of 2MPa.

Table 3-4 Static toughness of bio-board

	A2	B2	C2	D2	E2
Range (KPa)	23~91	64~106	47~109	53~99	66~124
Average (KPa)	48.3	76.5	67.5	74.0	85.0

3.4 Conclusion

In this chapter of research ten bio-boards were made with five experimental conditions and strength test were carried out to investigate their mechanical properties. The conclusions shown are as follows:

1) The results indicate that under all experimental conditions, making boards using

raw materials of corn straws was successful. Therefore, the board making process in this research is feasible.

2) In the bending test and tensile strength tests, the results showed that the stress increased with the increase of deformation for all Bio-boards. The stress reached a maximum value when the specimen was failed, then stress decreased sharply finally to zero.

3) The rupture stress varied in the range of 21.25MPa ~ 30.78MPa in the bending test. On the other hand, rupture stress range of 4.49MPa ~ 15.15MPa appeared in the tensile strength tests. Under the condition of 8MPa, bio-board has maximum bending strength as high as 29.37MPa. In tensile strength, the highest rupture stress of 10.89MPa was resulted in the pressure of 10MPa.

4) The result of stress-strain curves of tensile strength test reveals that the average range of Young’s modulus of bio-board is 1.4GPa~1.8GPa. Static toughness is larger as pressure becomes higher. With 10MPa pressure bio-board has maximum static toughness 85KPa.

The basic mechanical properties of bio-board were investigated and the results proved that bio-board could be created for use a packaging material, for heat insulation in architecture, and as a mulch film for agricultural purposes.

Chapter 4. Relation Between Refining Degree in grinding process and Size of Fiber

4.1 Introduction

This study will consider ways to improve the strength of bio-board for the purpose of exploring various uses of bio-board. In chapter 3, the effect of pressure applied in the forming process on the strength of bio-board was investigated. However, the results indicated that the resulting strength of bio-board was not significantly influenced by different pressures applied during the forming process. In addition, bio-board is a composite, thus, its strength is affected by numerous uncertain factors. According to previous research on bio-board making, many factors affect the strength of bio-board such as pressure, temperature, moisture content, length of fiber and so on.

In the last chapter, it can also could be composed that with different degree of refining, fiber were separated into several fractions, thus, satisfied size of fiber could be obtained using specific screens. However it is known that plant fibers which belong to lignocellulloses have complicated structure. Cellulose fibers are also divided into several varieties, such as seed fiber, phloem fiber, stem fiber and so on. Herbaceous fiber coming from corn straw is a sort of phloem fiber according to Lee (2007). Therefore, it is still need further investigation about whether or not could fine size distribution of fiber be obtained using specific screen.

In this chapter, corn straw will be used as an experimental sample and refined by a blender machine. Obtained corn straw pulp is divided into four grades according to the size of screen. After refining process, fiber size distribution of corn straw will be measured and investigated by a sampling survey. Finally we will compare the relationship between refining degree and fiber size' distribution.

4.2 Materials and methods

Sweet corn (Zea mays L. convar. mays) straws were used in this work. Corn straw was harvested on August 10th, 2013 (Liu 2009) at the Mie University Bio-resource

Department's experimental farm. After harvesting, grains were removed. Stem and leaves were left in a ventilated storage air-dried for two months.

During pretreatment, dry corn straws were cut into chips using an electric cutter, then soaked in water at 22℃ for 168 hours for softening the straw fiber. Figure 4-1shows the state of corn straw pulp before and after refining.

before refining after refining

Fig. 4-1 Corn straw pulp

According to the experimental condition shown in table 4-1, soaked straw were then fiberized (pulped) by using an atmospheric refiner with conical blades (Model A Beatfiner. Satomi. Corp.). The motor capacity is 11kw×4p-200, 60Hz, rotational speed is 1750 r.p.m (60Hz). The maximum flux control is 0.05-0.1m3/min. Air pressure is 0.6MPa required. Grinding part is an assembling conical cutter with blades. Dimension of cutter is 2.5mm×3.0mm×8° (blade width × slot width × blade angle).

During grinding process, fiber bundles would be fiberized by milling. Accordingly, milled-corn straw was sieved to possess particle size using a screen with the hole size of 2mm×2mm and a sieve with hole dimension of 0.2mm×0.5mm.

Table 4-1 Experimental condition for fiber collection

NO.	Sieve size (mm)	Refining time (min)
A1	0.2	5
A2	2	
B1	0.2	10
B2	2	

After grinding process, 10ml corn straw pulp was taken from four samples named A1, A2, B1, B2, where there are approximately 80 individual fibers. Thus, four samples containing 320 fibers were observed under a digital microscope (YAHATA shotto company, Japan) shown in figure 4-2. Specification of microscope is 100-120V, 50Hz, 150W. The lamp is 12V, 100W (JCR12V100W10H).

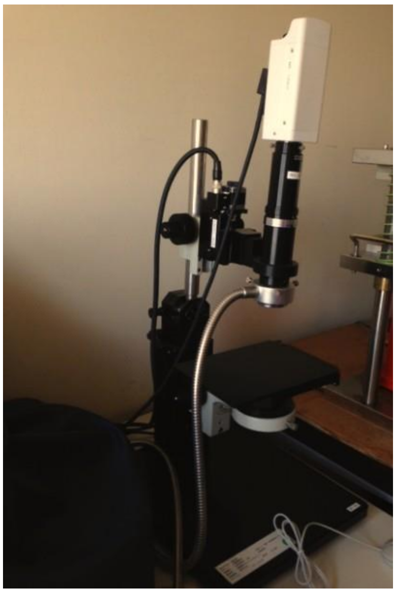


Fig. 4-2 Digital microscope

First, 10ml's fiberized corn straw pule which is from each sample is displayed on a glass without extra water content. Only refined fibers are placed on glass plate. Then arrange fibers separately using a tweezers, therefore, length of single fiber could be measured and the appearance of fiber is observed clearly under the digital microscope with the image processing software Focus-3D Vision FCS3D-MX.

4.3 Discussion

320 corn straw fibers were observed under a digital microscope, then, the length of each fiber was measured after using the software Focus-3D Vision FCS3D-MX. Two Images of single refined fiber taken by digital microscope are illustrated in figure 4-3 and figure 4-4. In the first image, there are two corn straw fibers with different width and length. Also, the edge of fiber is not smooth for some scrappy fibers still adhere to the main fiber. In the image of a single corn straw fiber shown in figure 4-4, two cellulose fibers are clearly observed. Cells can be seen in the fiber which is on the top because the outside layer is already removed by grinding. It also shows that a single refined corn straw fiber contains several cellulose fibers which are refined for the recombining in next forming process. In addition, it can be seen that the two cellulose fibers were broken as the end of fiber was with a rough edge.

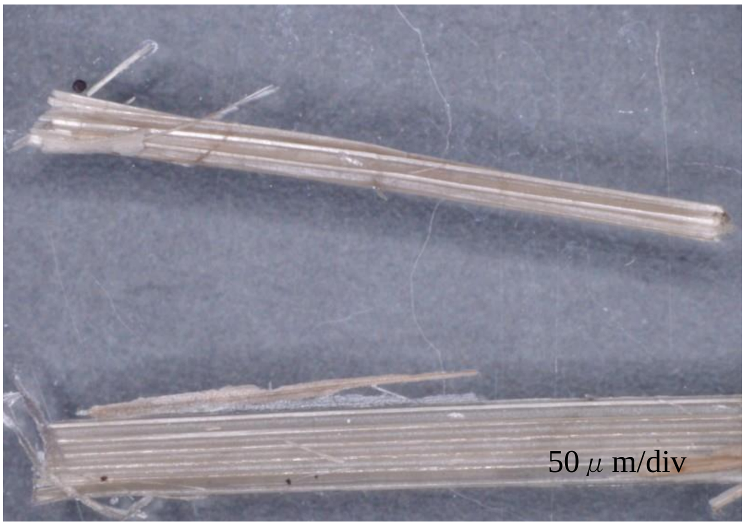


Fig. 4-3 Image of refined corn straw fibers

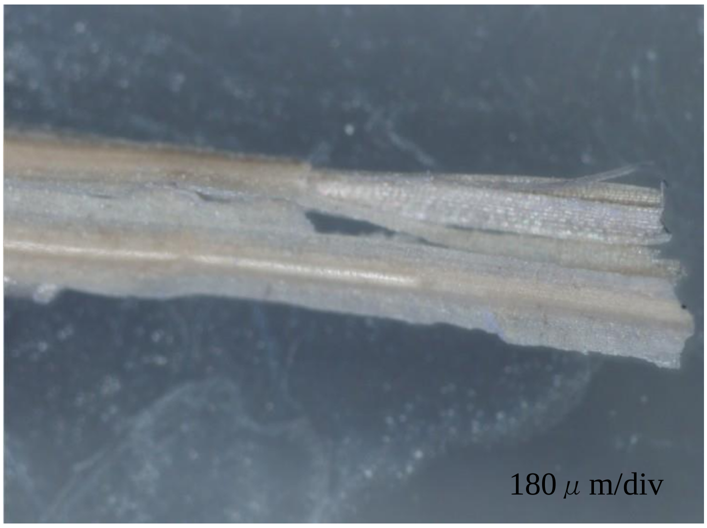


Fig. 4-4 Image of a single refined corn straw fiber

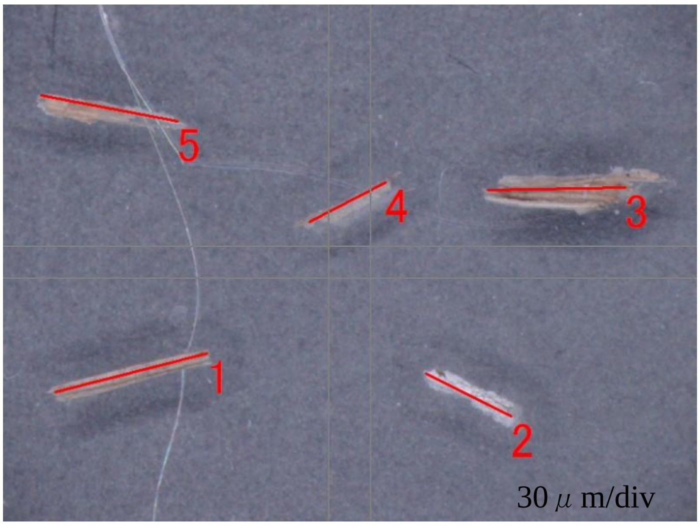


Fig. 4-5 Fiber length measurement

Fiber measurement is conducted as the picture shows in figure 4-5.

Fiber which is drop through filter was measured and the results were shown in figure 4-6. The distribution of fibers showed that in the same refining time of 5 minutes, A1 whose fiber is collected by a 0.2 mm-hole-sieve mainly distributed in the range of 1.5~6mm, for the other A2, the fiber distribution ranges from 4mm to over 10 mm. From these two kinds of fiber distributions, it can be known that a large fiber distribution in short size was obtained using a small size sieve, on the other hand, the bigger the sieve's hole is, the longer fibers were obtained.

It also can be found that fibers were not refined with ideal uniform dimension as the fiber with the length which is more than 4mm were existed in A2 and B2. In another words, corn straw fiber were not refined individually that fiber bundles still remained..

Furthermore, with the refining time of 10 minutes, fibers were divided into two varieties which is in the range of 0.5mm~2mm and the other is over 4mm. Therefore, using different size of sieve can collect different grades of length of fiber.

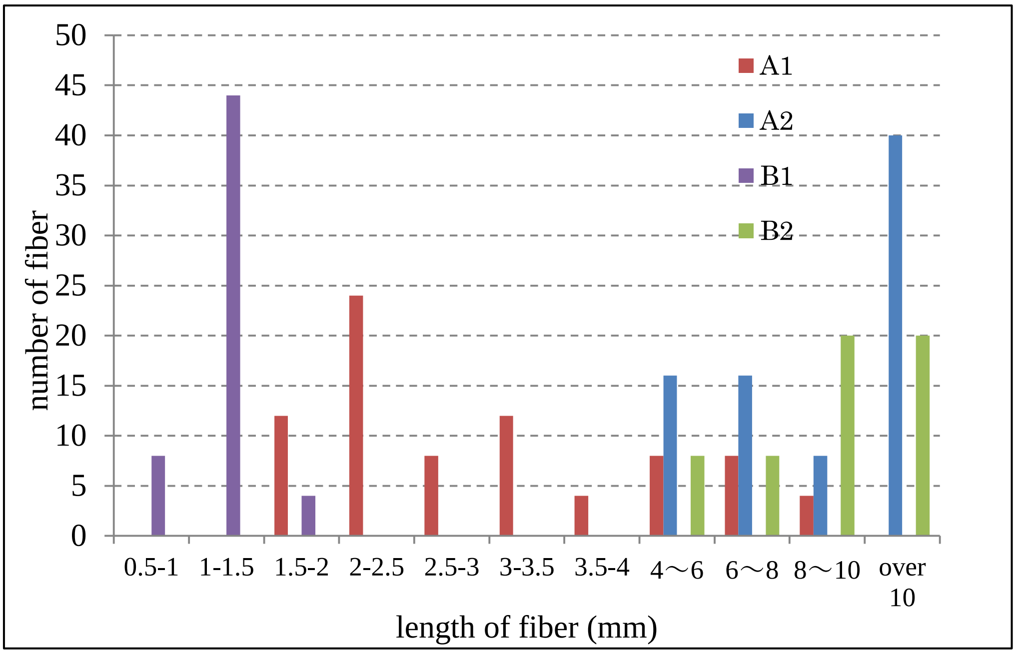


Fig. 4-6 fiber* distribution after grinding process

*fibers dropped through filter hole and were gathered to measure the size.

4.4 Results

1) Corn straw was ground into small dimension and the size of refined fiber length is significant different. Several cellulose fibers could be contained in one refined corn straw fiber and some cellulose fibers were broken.

2) Short fiber which is 0.5mm~2mm is mainly collected by 0.2mm-hole-sieve and long fiber which is over 4mm is significantly obtained by 2mm-hole-sieve. With the refining time of 10 minutes, fiber dimension were divided into two grades, however, corn straw fiber were not refined individually that fiber bundles still remained in A2 and B2.

4.5 A experiment for effect of refining degree on the strength of bio-board

As the basic principle of biomass material research, hydrogen bonding between cellulose fibers played a major part in the boarding making process. It is still unknown whether or not fiber bonding force is a valid consideration for determining bio-board strength compared to hydrogen bonding force. Paper made from agricultural fibers has lower tear strength (Schellenberger, 1995) and Ye (2007) also found that agricultural wheat straw fiber which is made of MDF (medium density fiber-board), resulted in a significantly higher modulus of elasticity. Ye's study revealed that using wheat fiber had significant effects on the strength of MDF.

Corn is a major crop in a number of countries, and, thus, corn straws are considered a good fiber source for low grades of paper. Here, we also consider corn straws as an ideal raw material for making bio-board. Corn straws are similar to sugarcane in structural features with an average fiber length of 1.5mm (0.5mm-2.9mm) and an average fiber width of 0.018mm (0.014mm-0.024mm). Typical fibers are fairly narrow, thick walled and have blunt or pointed ends, according to Ilvessalo-pfaffli (1995). The survey from Ilvessalo-pfaffli's survey suggests if corn straw fiber is refined between 0.5mm to-2.9mm, the main bonding force maybe depend on fiber bonding. On the other hand, if the length of fiber is less than 0.5mm, hydrogen bonding force may be the primary bonding force.

In this chapter, we will discuss how the length of fiber or, in other words, the degree of refining in the grinding process affects the strength of bio-board. First, in our study, boards were divided into test groups of two different lengths of corn straw fibers. Then, strength test were conducted on the boards. Finally, other mechanical properties, such as the Modulus of Elasticity (MOE), modulus of rupture stress (MOR) and static toughness (ST) were compared using the boards with two different fiber lengths.

4.6 Materials and methods

4.6.1 **Board making experiment**

Sweet corn (Zea mays L. convar. mays) straws were used in this work. Corn straw was harvested on August 10th, 2013 (Liu 2009) at the Mie University Bio-resource Department's experimental farm. After harvesting, grains were removed. Stem and leaves were left in a ventilated storage air-dried for two months.

Five processes applied in previous experiment are used in the present study which are cutting, soaking, grinding, compressing and drying showed in figure 4-7.

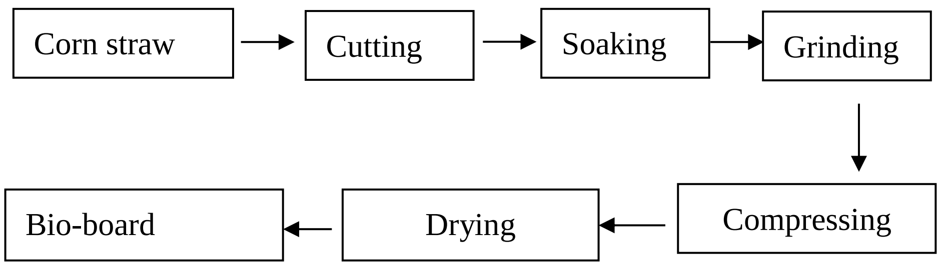


Fig. 4-7 flow chart of board making process

During pretreatment, dry corn straws were cut into chips using an electric cutter, then soaked in water at 22℃ for 168 hours for softening the straw fiber. In soaking process, corn straw fiber bundles absorbed moisture from water condition. It is easier to soften fiber bundles in a wet condition than destroy the structure of lignocelluloses fibers in a dry state. Soaking process is a preparation to the fiberization of corn straws. Soaked straw was then fiberized (pulped) by using an atmospheric refiner with conical blades (Model A Beatfiner. Satomi. Corp.). The motor capacity is 11kw×4p-200, 60Hz, rotational speed is 1750 r.p.m (60Hz). The maximum flux control is 0.05-0.1m3/min. Air pressure is 0.6MPa required. Grinding part is an assembling conical cutter with blades. Dimension of cutter is 2.5mm×3.0mm×8° (blade width × slot width × blade angle).Fiberization of corn straws at atmospheric pressure was carried out by passing the damp cut straw along with running water through the refiner' rotating blades. During grinding process, fiber bundles would be fiberized by milling. Accordingly, milled-corn straw was sieved to possess particle size using a screen with the hole size of 2mm×2mm and a sieve with hole dimension of 0.5mm×0.5mm. Photo of screen is shown in figure 4-8. Ground straws were fractionated into two fractions which possessed particle size of 0.5mm~2mm and the one which is longer than 2mm. The short type fibers which were collected by using SF type filter with the particle size of 0.5mm is named SF (short fiber). On the other hand, long type fiber LF (long fiber) is collected by LF type filter with particle size of 2mm. The flow chart of fiber collection is shown in figure 4-9.

SF type
particle size 0.5mm

LF type
particle size 2mm

Fig. 4-8 Types of screens

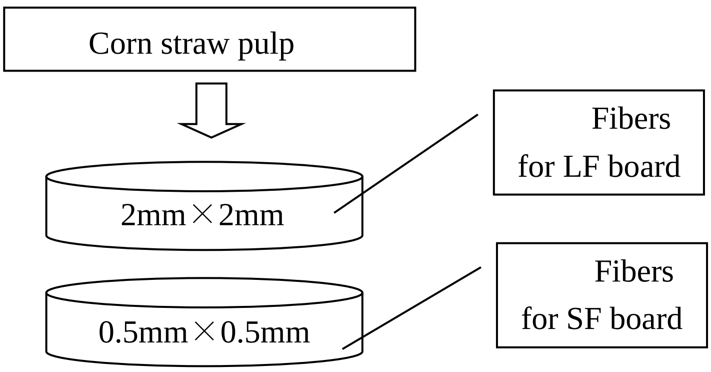


Fig. 4-9 Flow chart of fiber collection

A closed stainless steel die with some accessories including metal block, meshes, were designed which enabled obtaining one square board, 100 mm long ,100 mm width and 40mm depth. The calculated amount of ground corn straw was carefully filled in the die, and prepressed by for pressing excess water out from the die. Holes were drilled in the bottom of die, metal block and plate 2 mm in diameter, in a 7 mm×7 mm grid allowing water to escape in the forming process.

The desired pressure was applied at the maximum temperature of 110 ºC. It took 8-10 minutes until the die containing the samples reach the maximum temperature. Forming experimental conditions are displayed in table 4-2

Table 4-2 Board making experimental conditions

bio-board No.	pressure (MPa)	dying temp. (℃)
LF1,SF1	2	110
LF2, SF2	4	110
LF 3, SF3	6	110
LF 4, SF 4	8	110
LF 5, SF 5	10	110

4.6.2 **Strength test**

Bending tests and tensile strength tests were conducted for the purpose of analyzing the mechanical properties of bio-board. Specimens cut from one board were prepared for both bending test and tensile strength test. Figure 4-10 shows the detail of specimen division with a supersonic wave cutter which is used in specimen preparation.

In the Three-Point Bending Test, ten bio-boards named LF1 to SF5 were trimmed to 50mm×20mm×1.2mm. 40 rectangular beam specimens following standard JIS procedures and recommendations (JIS Z2248:1996) were prepared. All dimensions were measured with an accuracy of + 0.02 mm. Capacity of 100 N load cell was fixed on a motor, applied at a uniform rate of 0.57 mm/s in its vertical direction. Furthermore, the deformation signal was measured by a potentiometer. Both signals of force and deformation were transmitted into an amplifier and A/D convertor then logged in a computer. The bending stress of bio-board was obtained by the quotients of bending moment and section modulus of the specimen. Rupture stress was defined by quotients of maximum bending moment and section modulus of specimen when the specimen was fractured. The classic formula for determining the bending stress is:

$$\sigma_b = \frac{3PL_s}{2ba^2} \qquad (4.1)$$

Where P = force at fracture of test specimen; *Ls* = bearing distance between supports; b = width of test specimen; a = thickness of test specimen.

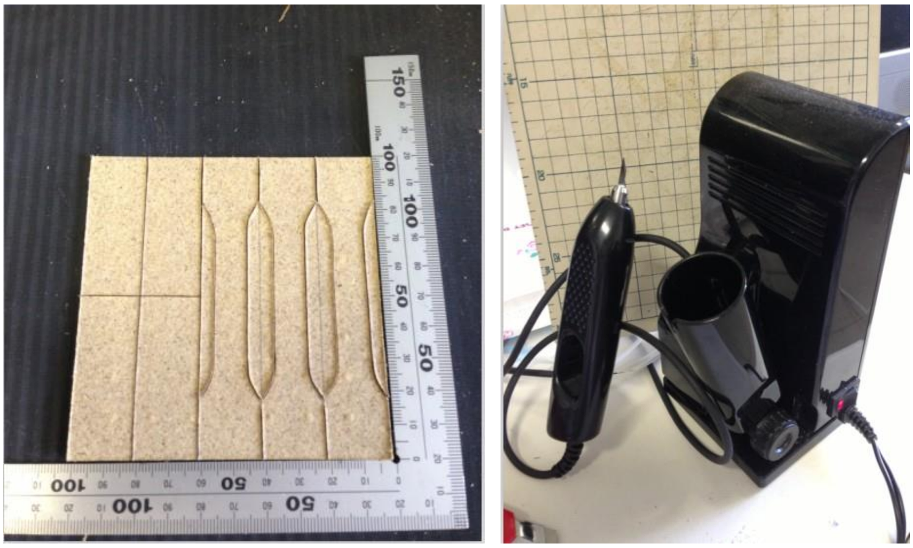


Fig. 4-10 Specimen division and a supersonic wave cutter.

To determine the internal bond strength, tensile strength tests were done under deformation control by using a universal testing machine. Speed of cross-head was 15mm/min. 30 specimens taken from ten bio-boards named LF1 ~ SF5 were subject to tensile strength tests following standard JIS procedures and recommendations (JIS z 2201).

Normal stress is defined by the quotients of axial load applied on specimen and original cross-sectional area of the specimen. Rupture stress was obtained when the axial load reaches to maximum value while the specimen was fractured. Rupture stress is expressed below:

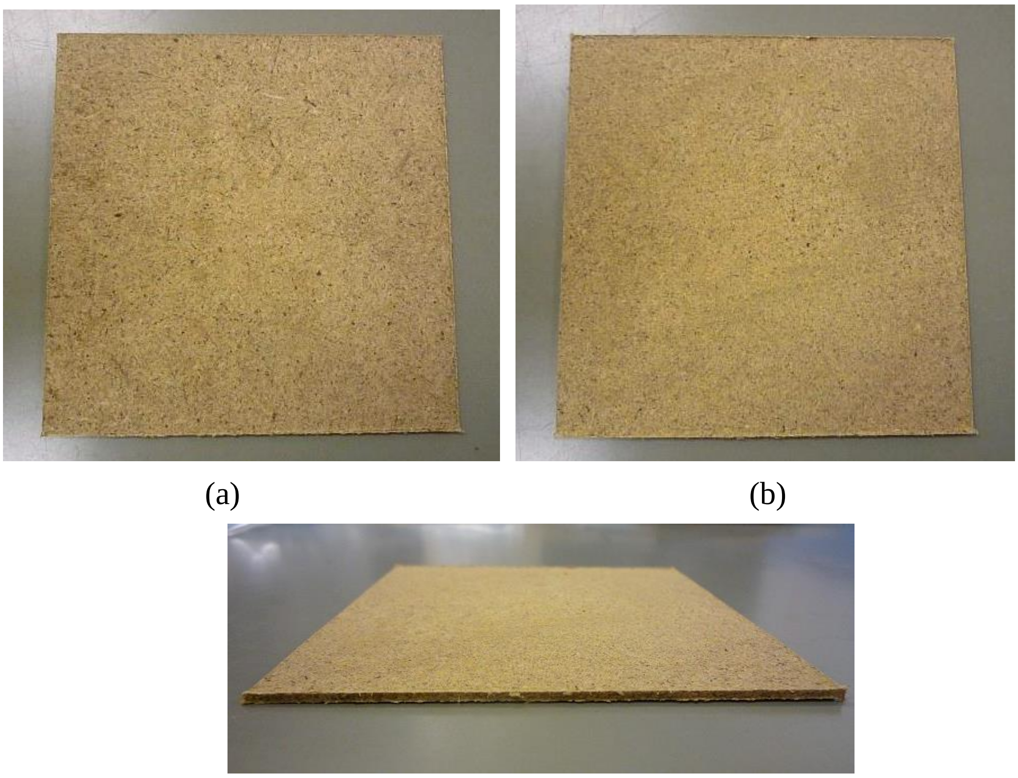
$$\sigma_t = \frac{P}{A} \quad (3.2)$$

Where P = the maximum axial load, A= original cross-sectional area of gauge section.

4.7 Results and discussion

4.7.1 **Board making**

40 specimens from ten bio-boards were subjected to the bending test and 30 specimens were used for the tensile strength test. The dimension of SF bio-boards were 100 mm×100 mm in area and in the range of 1.19 mm-1.36 mm in thickness and for LF bio-boards, the thickness is in the range from 1.15mm to 1.44mm. Figure 4-11 and figure 4-12 shows the LF and SF bio-board individually.



(a) (b) (c)

Fig. 4-11 Photos of LF bio-board (a: surface, b: bottom, c: section)

(a)

(b)

(c)

SF bio-board

Fig. 4-12 Photos of SF bio-board (a: surface, b: bottom, c: section)

It can be seen from the photos of LF and SF bio-board, the surface on which separate fibers are obviously observed in LF type, in the opposite, on both surface and bottom of SF bio-board, separate fibers could not be seen clearly. Two sides of board look similar. It is proved that fiber division operation is successfully done before compressing process.

Density of LF bio-board which is named LFB displayed in figure 4-13 indicates that bio-board has respective density in the range of 929kg/m^3-1050kg/m^3. Pressure which is from 2MPa to 10MPa increased with the increase of LFB density. For SFB density showed in figure 4-14, it ranges from 976 kg/m^3-1120kg/m^3. The trend of density variation is similar to LFB. The higher pressure the higher density was obtained. Density under 2MPa is the minimum. It changes to increase, however, with the pressures of 6MPa, 8MPa and 10MPa density does not increase significantly and almost become stable.

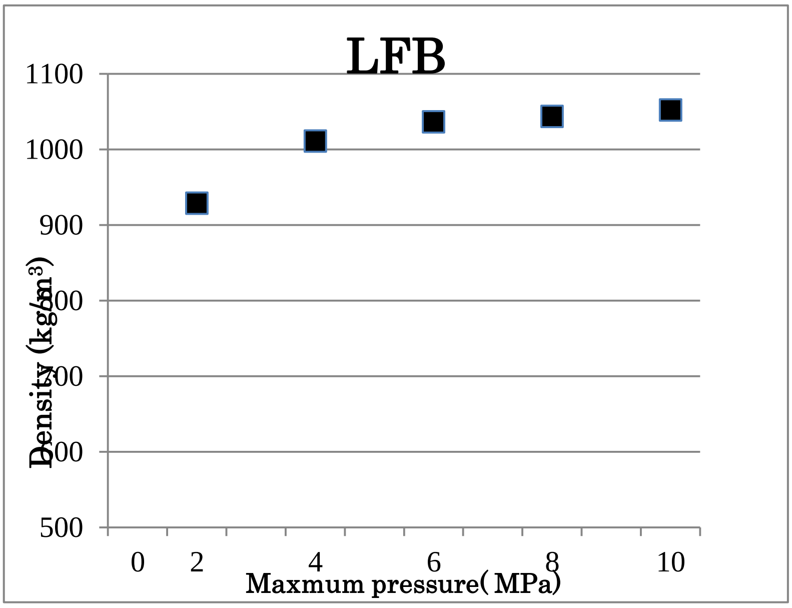


Fig. 4-13 Density of LFB

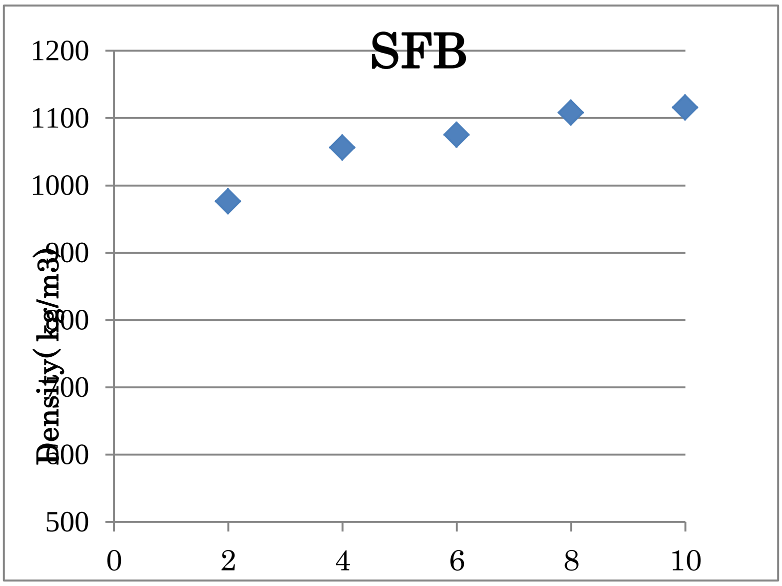


Fig. 4-14 Density of SFB

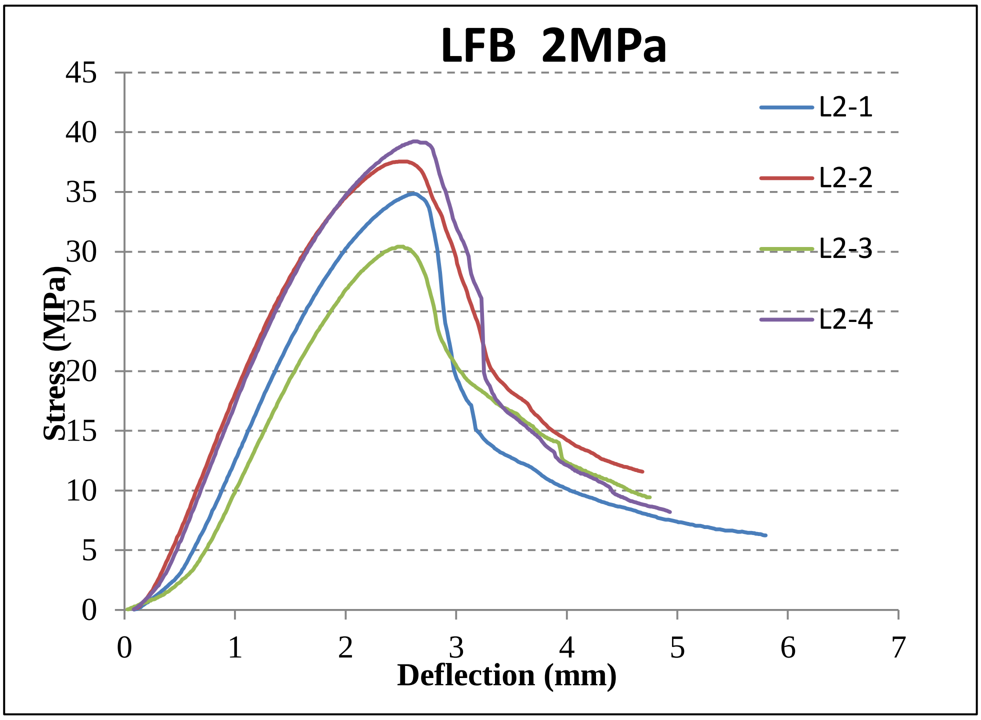
Moisture contents of LFB is in the range of 7.4%~8.2% and SFB made in this study showed a range of 6.4% ~ 8.0% in wet base. Properties of density and wet-basis moisture content of bio-boards are similar to MDF (medium density fiberboard) 5 Type ~ 30 Type based on JIS A 5905-2003.

4.7.2 **Bending strength test**

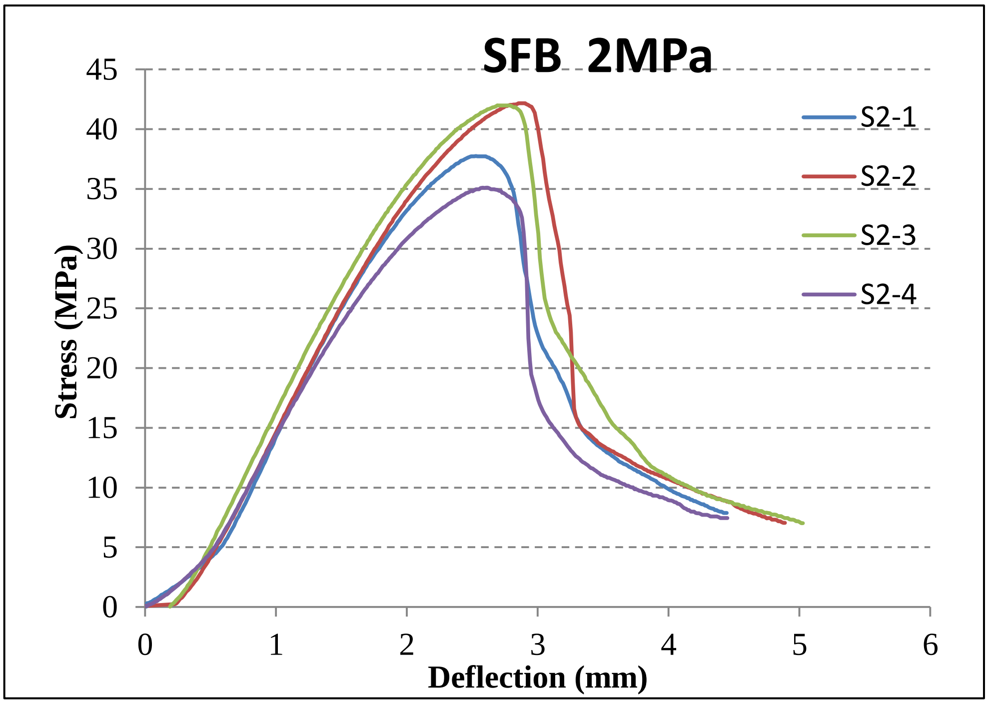
80 specimens obtained from board LF1 to board SF5 were provided for bending test.

The stress-deflection curves of ten boards are shown in figure 4-15. The changes of bending stress applied on four specimens of each bio-board are compared in the stress-deflection curves. Generally, stress value increased with the increase of deflection until reaching a maximum value. In addition, all the curves of stress-deflection almost approach a straight line before the fractures occurred. Specimens fractured at approximately 2.5mm of deflection. It implied bio-board has certain pressure resistance. Bending stress is called rupture stress when the specimen fractured. After specimen fractured, bending stress decreased sharply and is near to zero, however, bending stress still appears a few because some fibers were still remained connecting which happened in all specimens.

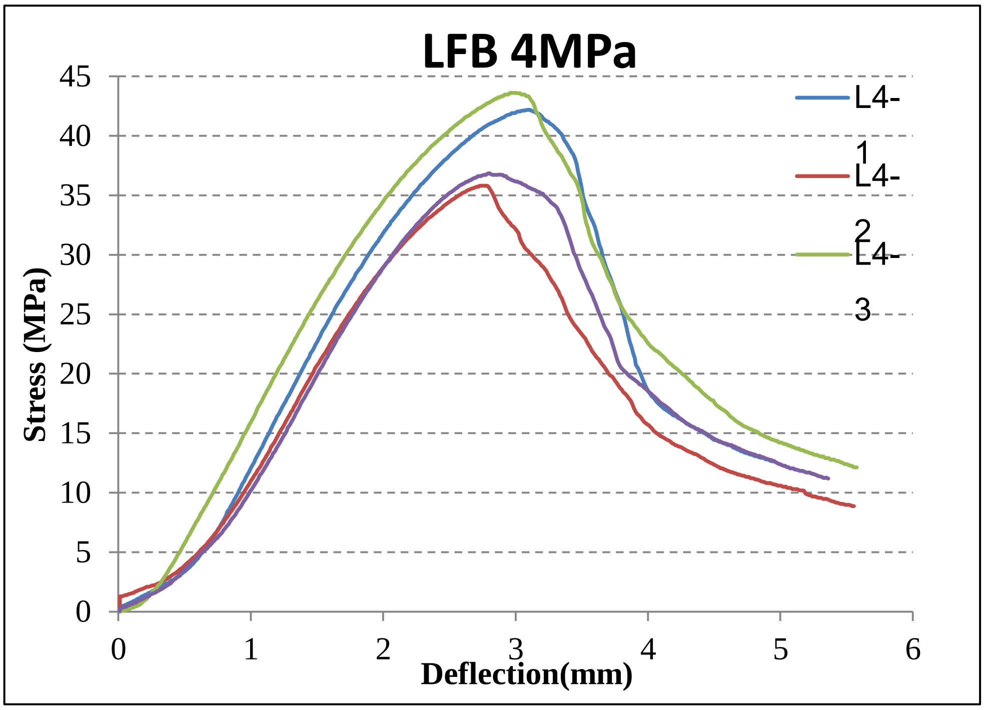
As bio-board are made of two lengths of fibers, under the same pressure applied in forming process, variation of stress between LFB and SFB shows slight different. In general, stress becomes greater as fiber becomes shorter. Under the pressure of 2MPa, 6MPa, 8MPa and 10MPa, maximum stress of SFB are greater than the one of LFB. With the pressure of 4MPa, although maximum value is in specimen of L4-3 which is from LFB, however, variation of fracture points from LFB is bigger than the one from SFB. It can be seen in SFB 4MPa, stress of 4 specimens are in the range of 35MPa ~ 40MPa, in the opposite, the stress of specimens in LFB with the same pressure 4MPa shows the range from 35MPa to 45MPa. Uncertainty could be considered to explain the stability of fracture points. As Mudit,C (1998) writes that the minimum fiber length necessary to produce acceptable paper strength properties is dependent on many factors, and fiber lengths are not unequivocally related to paper strength properties (Young, 1997). Different fiber lengths are desirable for different properties in paper. For example, longer fiber length is desirable for strength properties in paper, but they tend to bunch together and as a result do not provide good formation. Shorter fibers on the other hand provide excellent formation.



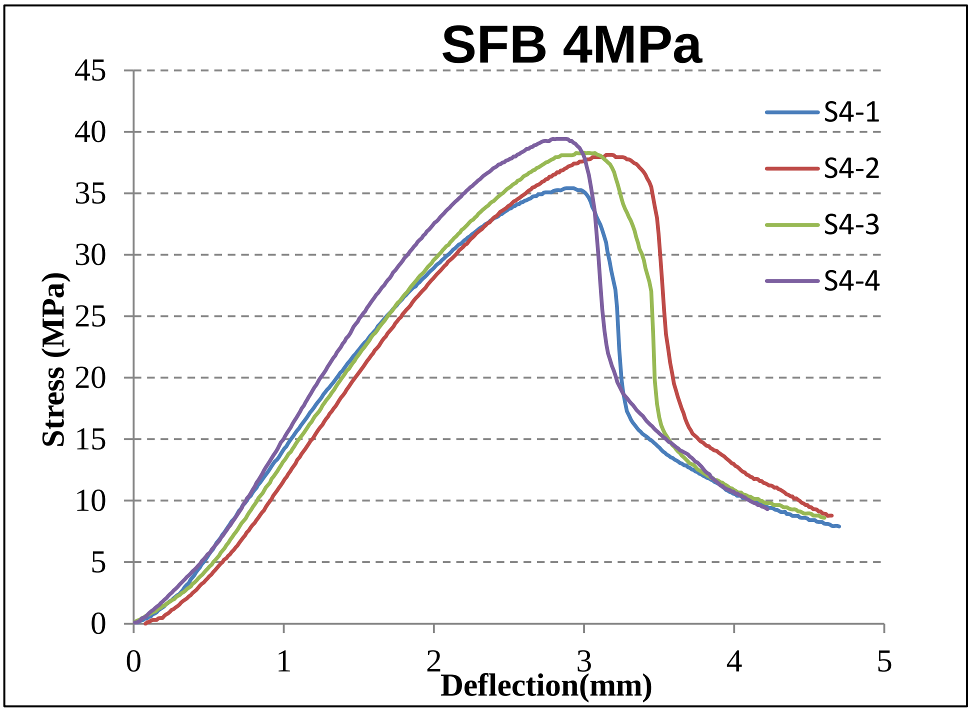
a. Stress-deflection curve of 2MPa LFB in bending test



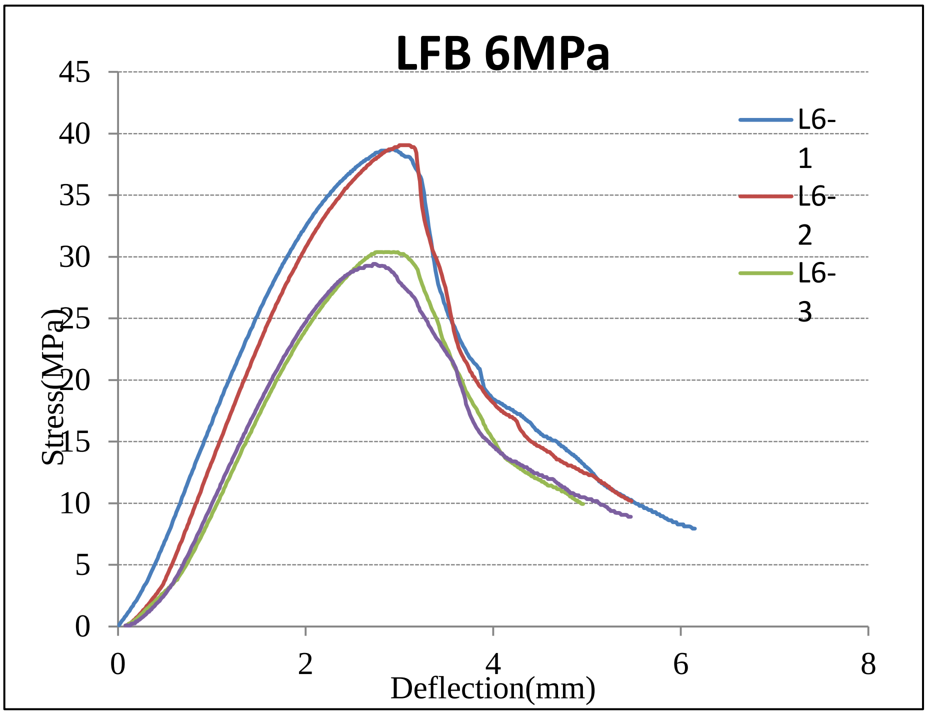
b. Stress-deflection curve of 2MPa SFB in bending test



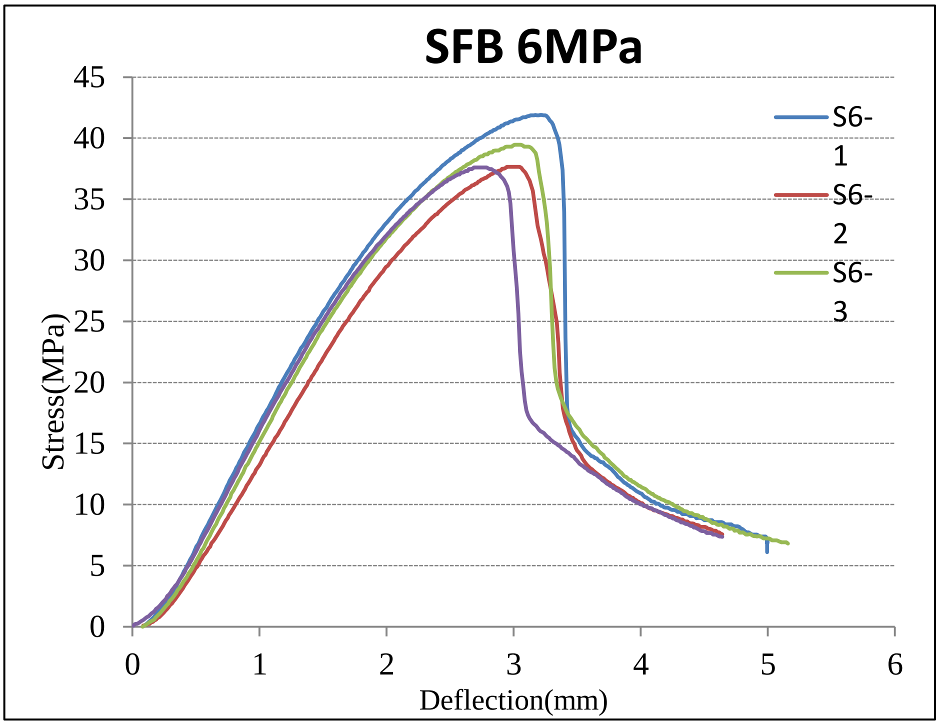
c. Stress-deflection curve of 4MPa LFB in bending test



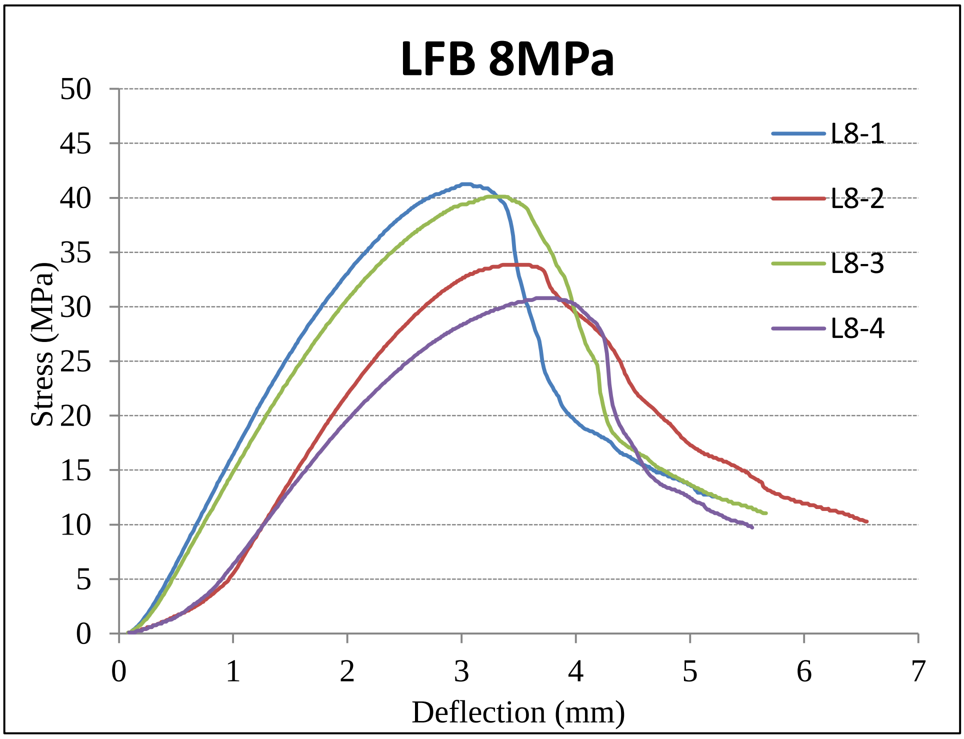
d. Stress-deflection curve of 4MPa SFB in bending test



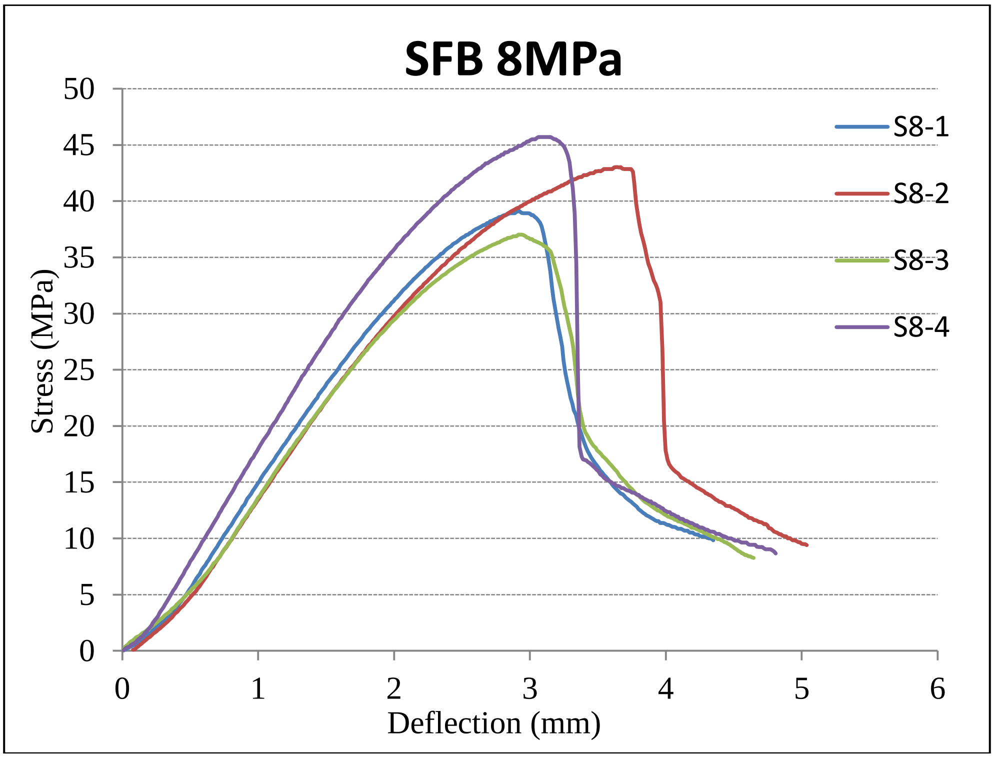
e. Stress-deflection curve of 6MPa LFB in bending test



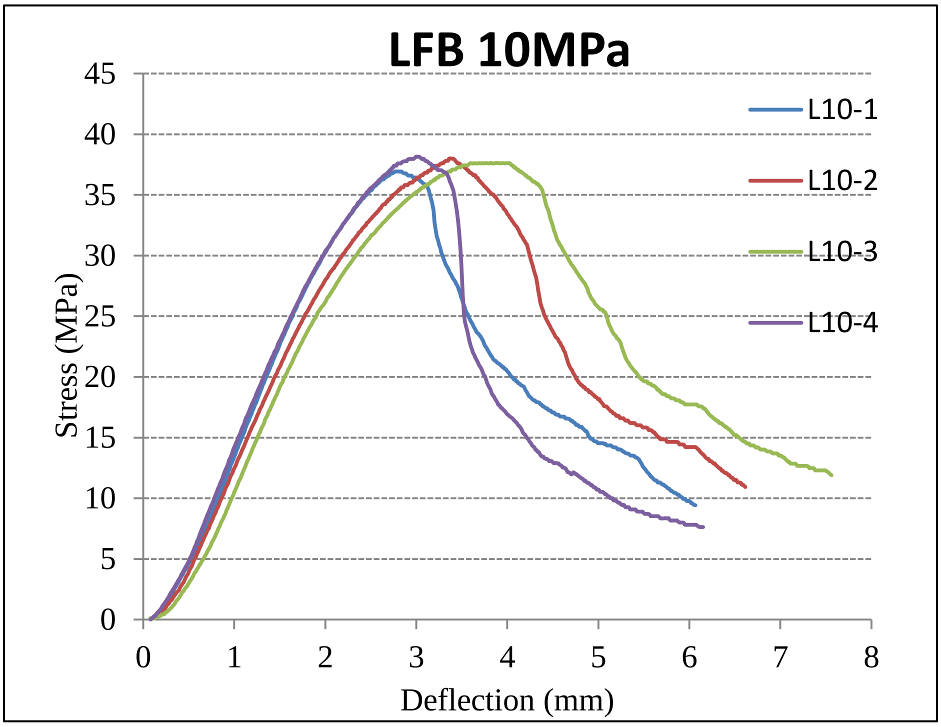
f. Stress-deflection curve of 6MPa SFB in bending test



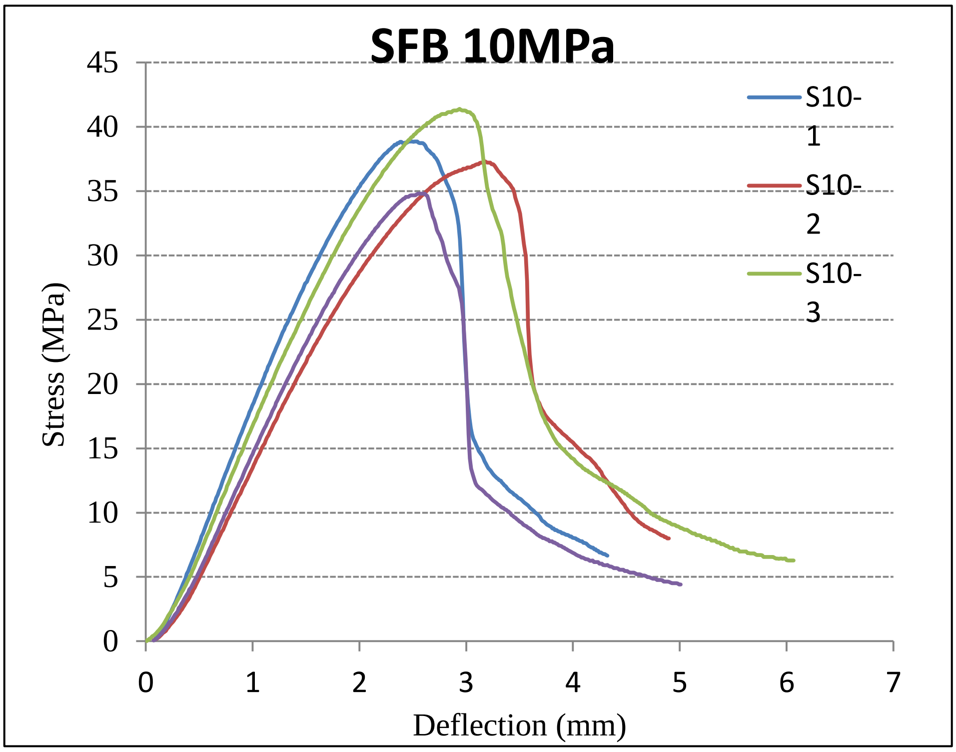
g. Stress-deflection curve of 8MPa LFB in bending test



h. Stress-deflection curve of 8MPa SFB in bending test



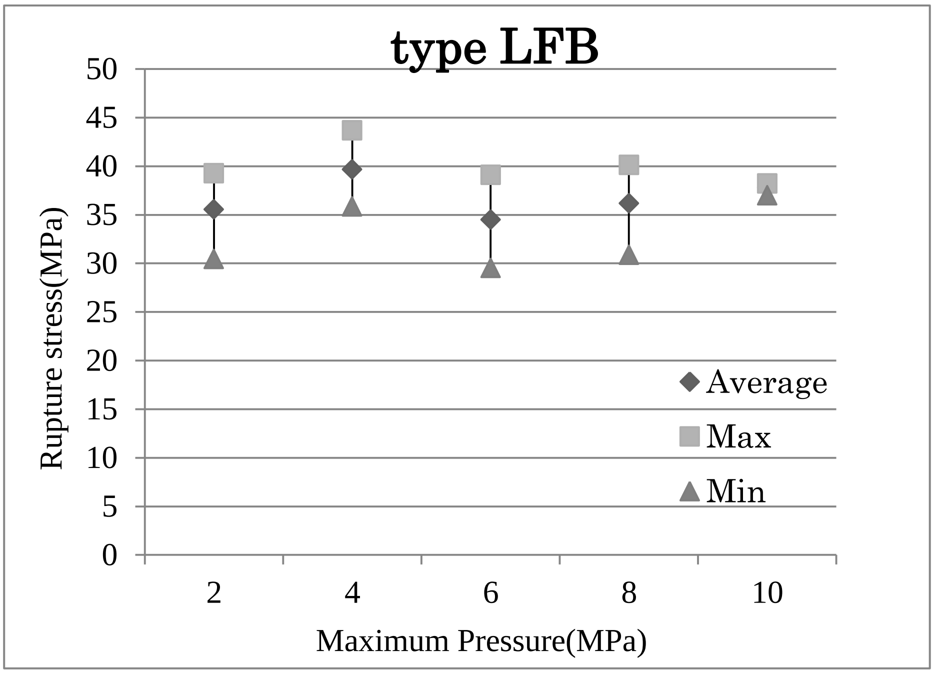
i. Stress-deflection curve of 10MPa LFB in bending test



j. Stress-deflection curve of 10MPa SFB in bending test

Fig. 4-15 stress-deflection of bio-board in bending test

In figure 4-16, different rupture stress values of bio-boards maximum value, average value and minimum value were calculated from stress-deflection curves. The results indicate that different rupture stress is obtained by different specimen which is even from the same board.



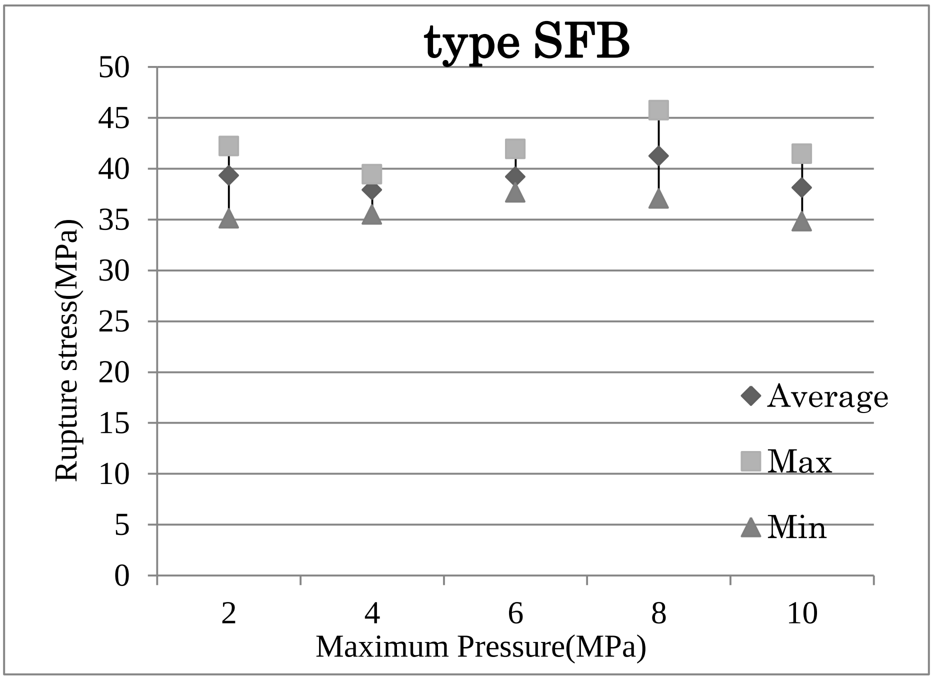


Fig. 4-16 Rupture stress of bio-board

Average of rupture stress from LFB and SFB in bending test are shown in table 4-4. The resulting of strength test implies that rupture stress of ten bio-boards is obviously different. The maximum bending rupture stress 41.25MPa occurred in SFB with the condition of 8MPa. The minimum bending rupture stress is in LFB with the pressure of 2MPa. It is found except 4MPa, under other pressure conditions, rupture stress of SFB is greater than the one of LFB. Therefore, results of bending strength test prove short fiber bio-board with 8MPa pressure is the optimum condition to make bio-board.

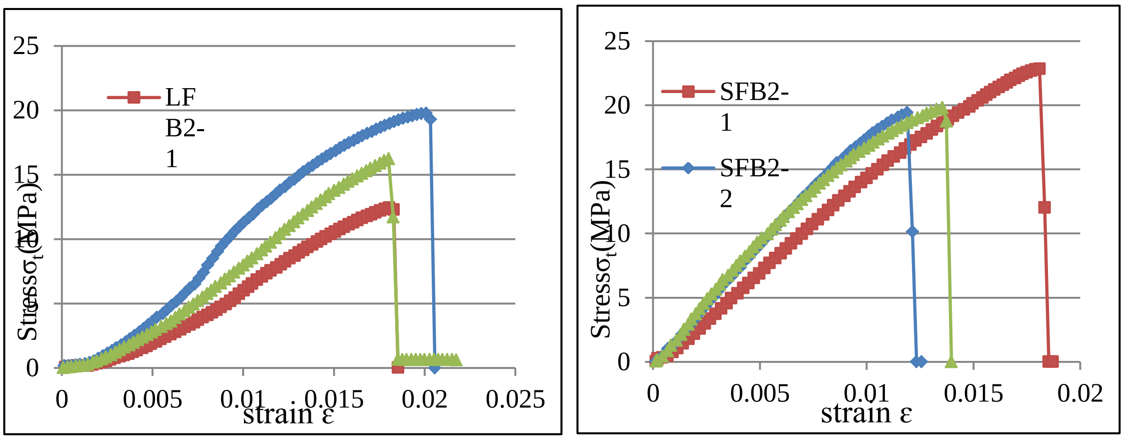
Table 4-3 Average of rupture stress from LFB and SFB in bending test

	2MPa	4MPa	6MPa	8MPa	10MPa
LFB ARS(MPa)	35.56	39.67	34.52	36.20	37.80
SFB ARS(MPa)	39.32	37.90	39.19	41.25	38.13

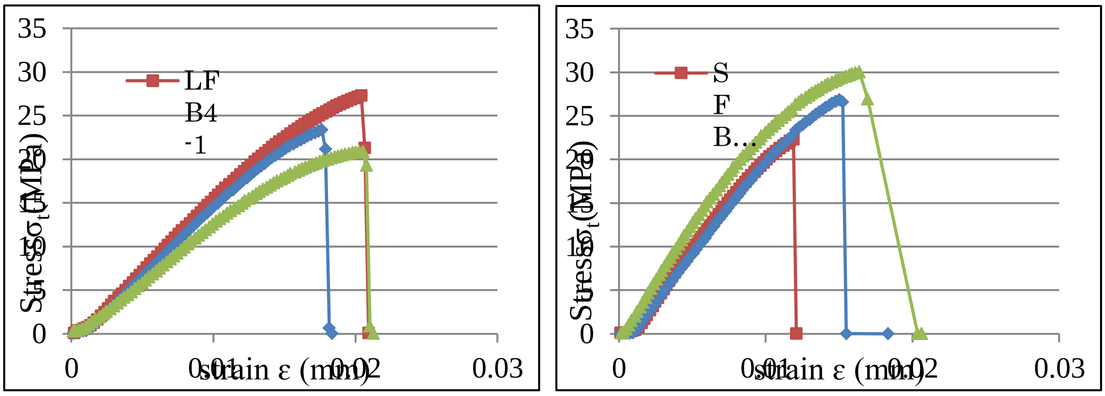
4.7.1 **Tensile strength test**

30 specimens were used in tensile strength test for the purpose of mechanical property investigation. First, the relationship between stress and strain were shown in figure 4-17 with ten graphs. For LFB and SFB are made with the same process, under each pressure condition (2MPa, 4MPa, 6MPa, 8MPa, 10MPa), the comparison of stress and strain can be seen obviously. It is found that in the beginning of all the stress-strain curve, tensile stress increases with the increase of strain, after the tensile stress reaches the maximum value, specimens were broken, then, tensile stress decreases suddenly to zero. The maximum value is called tensile rupture stress. According to the curves of stress-strain, it can be known that fracture points of 3 specimens taken from the same board are totally different. Thus, rupture stress were not the same which were showed in table 4-5. The maximum, minimum and average of rupture stress from LFB and SFB in tensile strength were shown in figure18-19. The rupture stress of LFB and SFB resulted that generally rupture stress of SFB is greater than the one of LFB.

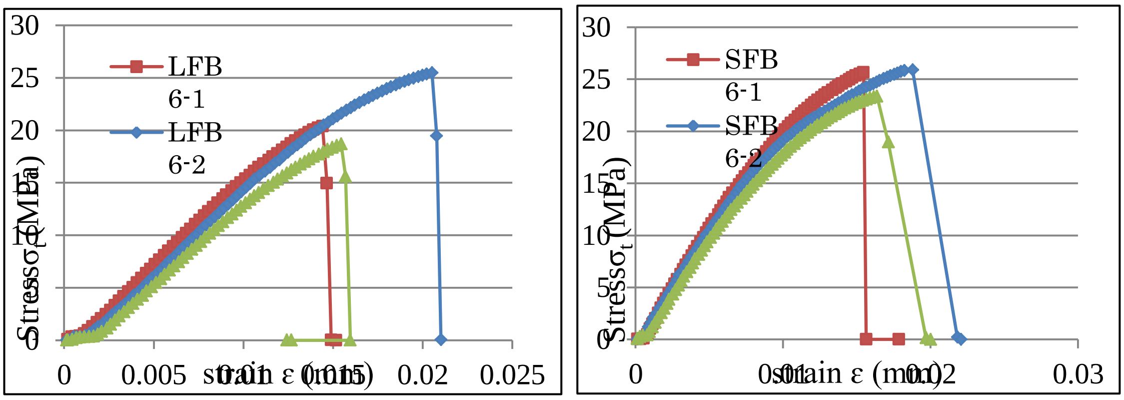
As the curve of stress-strain shows a proportional line before the point of fracture, the partial proportional curve was modeled and Young's modulus was calculated which were showed in table 4-6



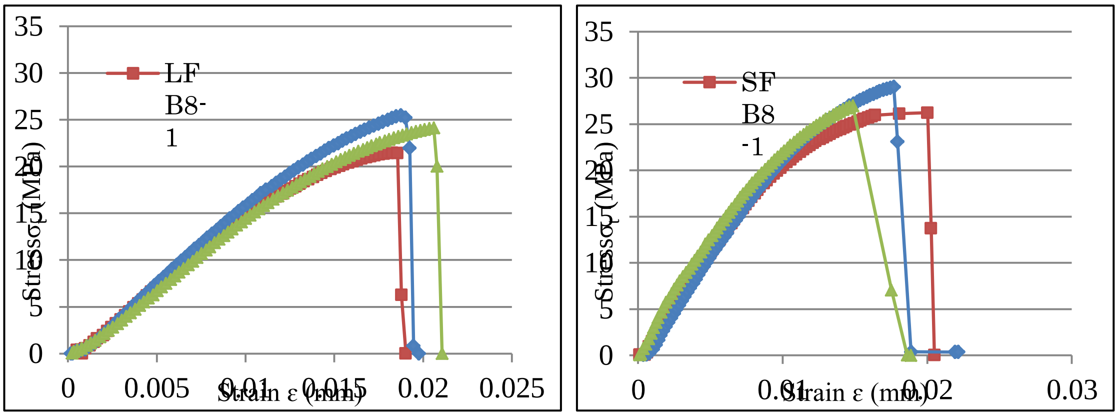
a. Stress- strain curve of LFB and SFB made with 2MPa



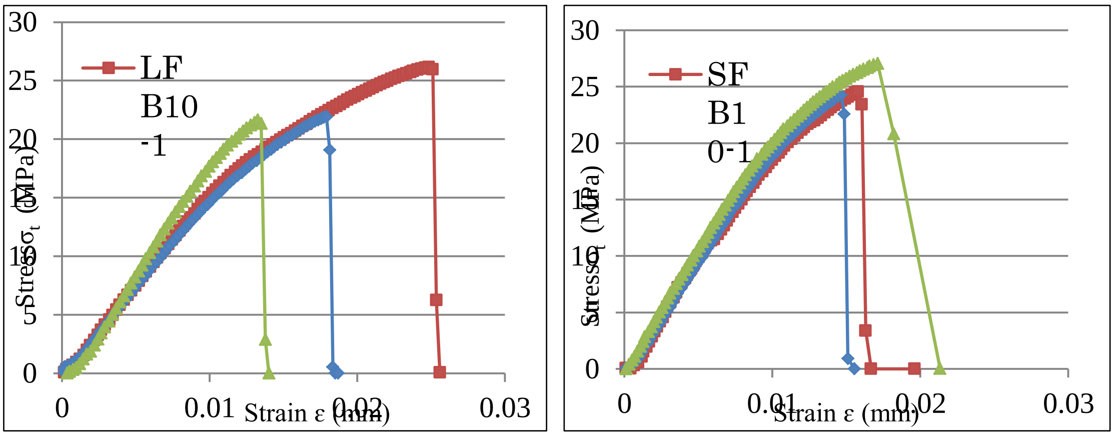
b. Stress- strain curve of LFB and SFB made with 4MPa



c. Stress- strain curve of LFB and SFB made with 6MPa



d. Stress- strain curve of LFB and SFB made with 8MPa



e. Stress- strain curve of LFB and SFB made with 10MPa

Fig. 4-17 comparison of stress-strain curve for LEB and SFB in tensile strength test

Table 4-4 Rupture stress of LEB and SFB in tensile strength test

	Long Fiber Board (LFB)				Short Fiber Board (SFB)			
Applied pressure(MPa)	P1	P2	P3	Ave.	P1	P2	P3	Ave.
2	12.43	19.75	16.23	**16.14**	22.83	19.44	19.82	**20.69**
4	27.27	23.33	23.33	**23.82**	22.31	26.86	30.02	**26.40**
6	20.35	25.45	18.70	**21.50**	25.65	25.90	23.36	**24.97**
8	21.45	25.48	24.12	**23.69**	26.23	29.03	26.97	**27.41**
10	26.15	21.91	21.63	**23.23**	24.57	24.29	27.06	**25.31**

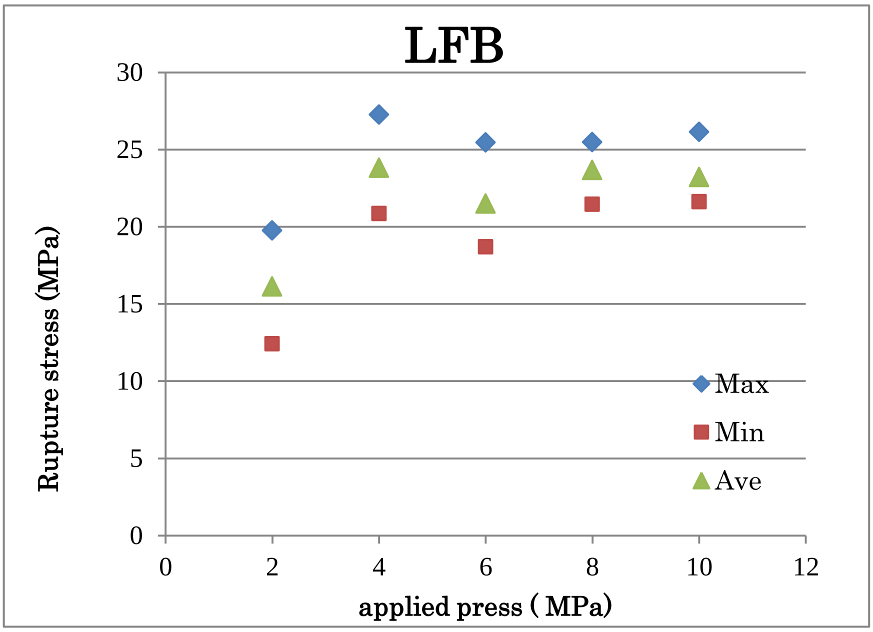


Fig. 4-18 Rupture stress of LFB in tensile test

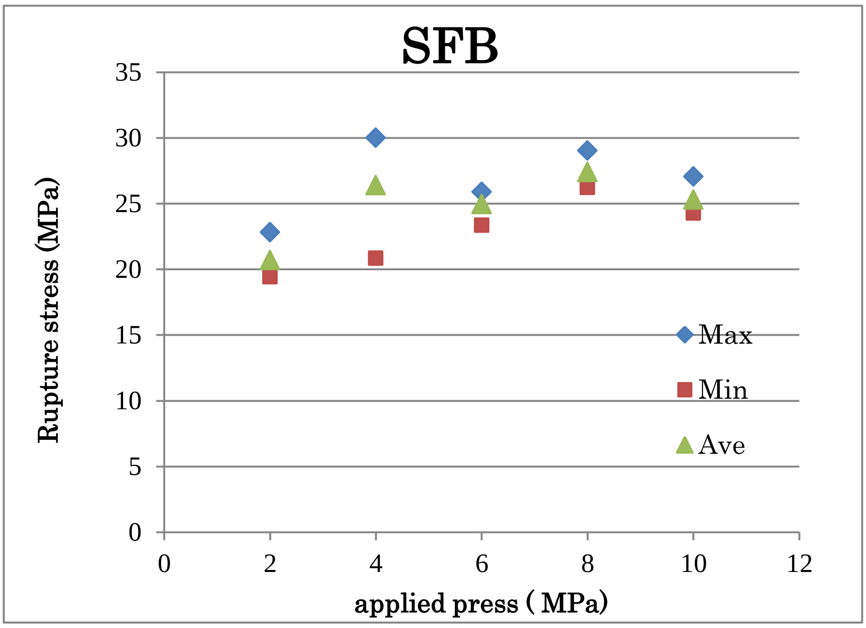


Fig. 4-19 Rupture stress of SFB in tensile test

It has been found that the range of E in every bio-board is different. 4 specimens of one board showed various deformation performances. The average range of Young's modulus of LFB is 0.86GPa~2.45GPa, on the other hand Young's modulus of SFB ranges from 1.59GPa to 2.01GPa shown in table 4-5.

Table 4-5 Average of Young's modulus in tensile strength test

	2MPa	4MPa	6MPa	8MPa	10MPa
LFB Ave.(GPa)	0.86	1.33	1.36	2.45	1.43
SFB Ave.(GPa)	1.59	2.07	1.73	2.01	1.80

Average static toughness of five bio-boards is displayed in table 4-6. The results found that the length of fiber has a great influence on the specimens. It affects rupture stress and also the static toughness. The results of static toughness showed that with the pressure of 8MPa LFB has the maximum static toughness 297KPa for the reason that a long fiber is with a greater elasticity property while a short fiber is easy to be broken by tensile force.

Table 4-6 Static toughness in stensile strength test

	2MPa	4MPa	6MPa	8MPa	10MPa
Range for LFB(KPa)	156~246	257~341	234~319	271~319	262~325
Average (KPa)	201	297	269	297	286
Range for SFB(KPa)	196~277	224~305	243~272	239~290	245~274
Average (KPa)	225	268	259	266	256

4.8 Conclusions

In this chapter, totally ten bio-boards which were divided into LFB and SFB were made with five experimental conditions. Strength test were carried out to investigate their mechanical properties. The conclusions shown are as follows:

1) The results indicate that under all experimental conditions, using two different length of fiber for making boards was successful. Therefore, the board making process

and conditions in this research is feasible.

2) In strength tests, the results showed that the stress increased with the increase of deformation for all Bio-boards in bending test. Also the stress is proportional with strain before it fractures in tensile strength test. The stress reached a maximum value when the specimen was failed, then stress decreased sharply finally to zero.

3) The average of rupture stress varied in the range of 34.52MPa ~ 39.67MPa for LFB. On the other hand, rupture stress range of 37.9MPa ~ 41.25MPa appeared in SFB in the bending test. In tensile strength, rupture stress varied in the range of 16.14MPa ~ 23.82MPa for LFB. On the other hand, rupture stress range of 20.69MPa ~ 27.41MPa appeared in SFB. The rupture stress of LFB and SFB resulted that generally rupture stress of SFB is greater than the one of LFB. Short fiber had more influence than longer fiber the strength of bio-board.

4) The result of stress-strain curves of tensile strength test reveals that the average range of Young’s modulus of LFB is 0.86GPa~2.45GPa meanwhile the average range of Young’s modulus for SFB shows a range of 1.59GPa~2.07GPa. There was not obvious variation between LFB and SFB by static toughness.

Chapter 5. Strength Comparison for Corn Straw Bio-board and Other materials

5.1 Introduction

Corn straw bio-board is known as a friendly environmental material which is biodegradable for the earth after being used. Because of strength test made in the previous experiments, which indicated that corn straw bio-board has certain strength to bear load. It could be considered to apply in packing materials potentially in daily life.

According to United States Environmental Protection Agency and American Chemistry Council, polymers abound in nature (2005). Rubber tree cellulose has been used as raw material to make manufactured polymeric and plastics. As many common classes of polymers, they are composed of hydrocarbons, compounds of carbon and hydrogen which indicated that cellulosic bio-board is an example of natural polymers.

In previous study, strength test was conducted and resulted that bio-board could bear certain load, however, bio-board which is applied in package needs some more investigations comparing with other packing materials. Polymer products package such as polystyrene food containers, corrugated cases, woody boxes are common used in super markets. The low cost and convenience of these package is popular and welcomed by people, however, environment pollutions still threaten the human beings, furthermore, woody resource is also decreasing yearly, because of which a new clear package is possibly needed.

In this chapter, four packing materials which are a piece of wood, polystyrene food try, package box for digital camera and a corrugated case were subjected to a strength test. Then, comparison of rupture stress and Young' modulus between corn straw bio-board and the four packing materials was made and discussed. The study of the other four materials' mechanical properties helps to understand the application range of bio-board.

5.2 Materials and methods

At first, a piece of cedar wood with the dimension of 10mm×250mm, a polystyrene food container, a paper box for a digital camera case and a corrugated case were prepared for strength tests. The four samples were named CW (cedar wood), PC (polystyrene try), PB (paper box), CC (corrugated cases). The four test samples were cleaned by removing dust from the surface. The film coating on PT was also taken off from the body, for bio-board was not covered by any coating materials. table 5-1 shows the dimension of specimens which were from the four materials.

Table 5-1 dimension and density of four samples

	CW	PT	PB	CC
Thickness (mm)	1.3~1.5	3.5~4	1.9~2	4.6~4.8
Width (mm)	11.6~11.8	9.3~10.1	10.1~10.8	24.8~25.25
Density of wet base (g/cm3)	*0.38	*0.925~0.94	*0.085×10^{-6}	*0.044×10^{-6}

*data of density is cited from Whitfield (1996), wood museum (2014), toishi.info (2014)

Bending test was carried out by a strength test machine shown in figure 3-4 of chapter 3 for investigating bending rupture stress of the four samples. In the Three-Point Bending Test, 3 specimens cut from CW, PT and PB were trimmed to 50mm×10mm(±0.2mm) beam respectively. Because the special structure which is called inner cushioning (Nakagawa, 2006) of CC that dimension of specimen for CC was made 2.5times larger than the other three materials.

All dimensions were measured with an accuracy of + 0.02 mm. Capacity of 500 N load cell was fixed on a motor, applied at a uniform rate of 15mm/min in its vertical direction. Furthermore, the deformation signal was measured by a potentiometer. Both signals of force and deformation were transmitted into an amplifier and A/D convertor then logged in a computer. The bending stress of bio-board was obtained by the quotients of bending moment and section modulus of the specimen. Rupture stress was defined by quotients of maximum bending moment and section modulus of specimen when the specimen was fractured. The classic formula for determining the bending stress is the same to corn straw bio-board bending stress calculation defined in chapter 3.

5.3 Results and discussions

4 individual specimens were applied in bending stress test and the variation of stress-deformation curves were compared as follows. figure 5-1 shows that stress of CW increased with the increase of deformation before the specimen fractured. A proportional liner could be found in the graph before the maximum point of stress. After specimen was ruptured stress decreased sharply, however some fibers still connected even specimen fractured. That is why between 2mm and 3mm the stress stopped decreasing. As the continue increase of deformation, stress fell down to zero which means specimen was fractured completely with certain deformation in bending strength test. The variation stress-deformation for CW was mostly similar to bio-board for CW belongs to wood and its cellulose content is approximately closed to corn straw.

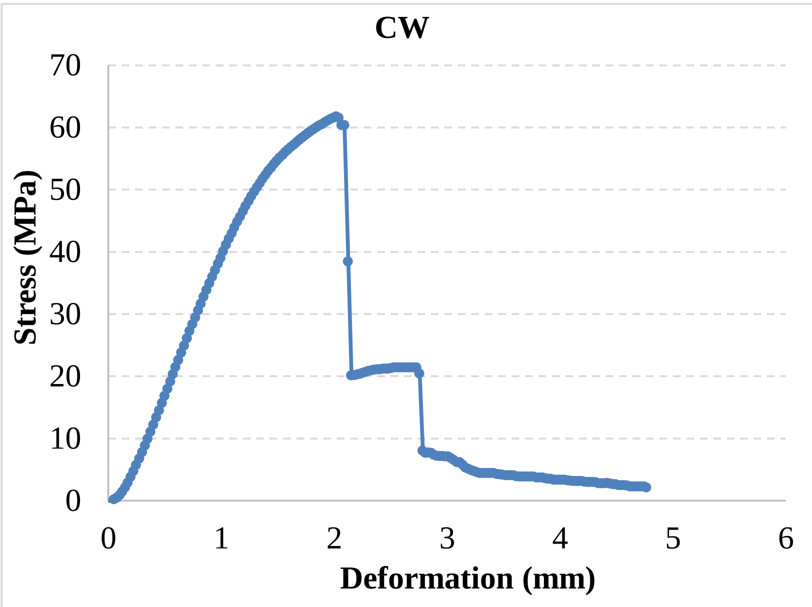


Fig. 5-1 Stress-deformation curve of CW in bending test.

In the other hand, figure 5-2 showed a quite different variation of stress-deformation curve of PT. It seems that there was not any obvious yield point in the variation of stress-deformation curve of PT, however, it could be found that before deformation was 1mm, the stress varied with deformation as a proportional liner, the curves' steepness was downward sloping after deformation was over 1mm. Deformation increased until a fracture occurred than stress became zero.

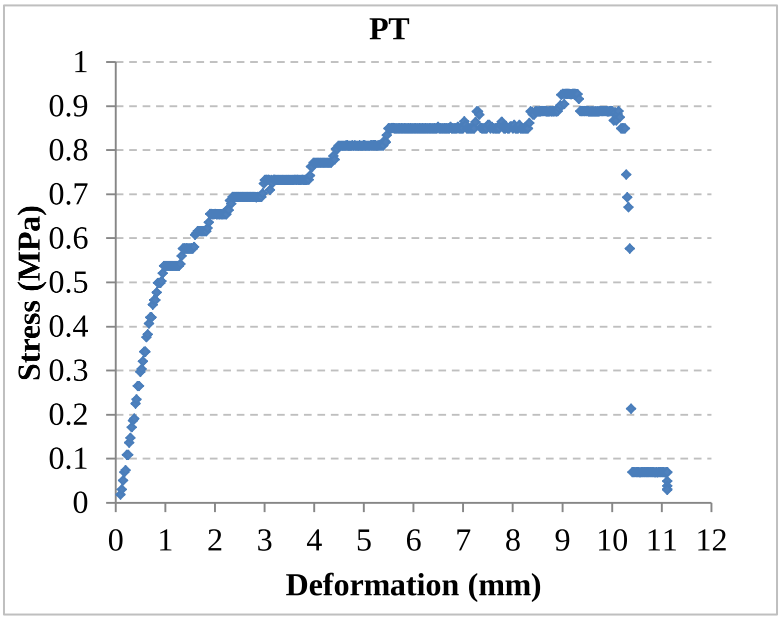


Fig. 5-2 Stress-deformation curve of PT in bending test.

For the variation of stress-deformation curve of PB shown in figure 5-3, the variation of curve differed slightly from CC, which was after specimen fractured, stress decreased, however, the decrease of stress stopped and kept constantly at approximately 3MPa. In the picture (c) shown in figure 5-5, it could be known that there

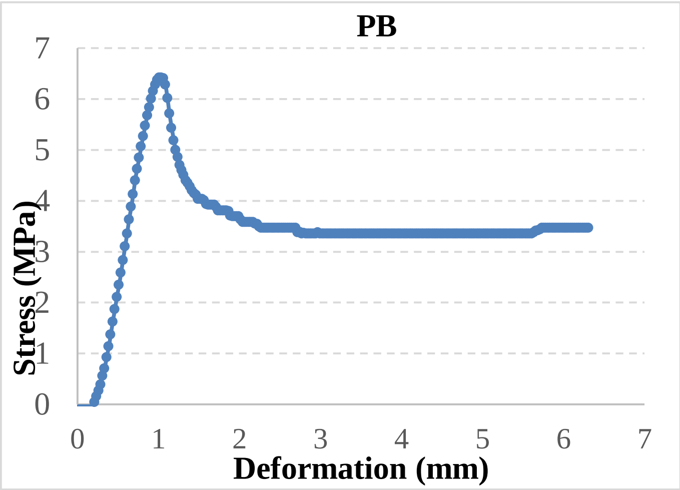


Fig. 5-3 Stress-deformation curve of PB in bending test.

was not any fracture cracks occurred in PB specimen, which means the specimen of PB failed but it was not completely broken by applying load. The behavior of the specimen was considered to be similar as the ductile material.

In the last stress-deformation curve of CC displayed in figure 5-4, a specific variation of stress and deformation was found. At first, before the specimen of CC yield, the trend of stress and deformation which was a proportional liner was similar to the other three materials. However, after over the yield point, stress decreased slightly and then increased again. It could be seen that with two peaks and valleys which reflected the trend of stress might continuously vary without falling down to zero, because there was not any fractural cracks happening in specimen of CC in figure 5-5 (d). This variation might be considered that peaks and valleys happened in stress variation was attributed to the cushioning properties of corrugated structures.

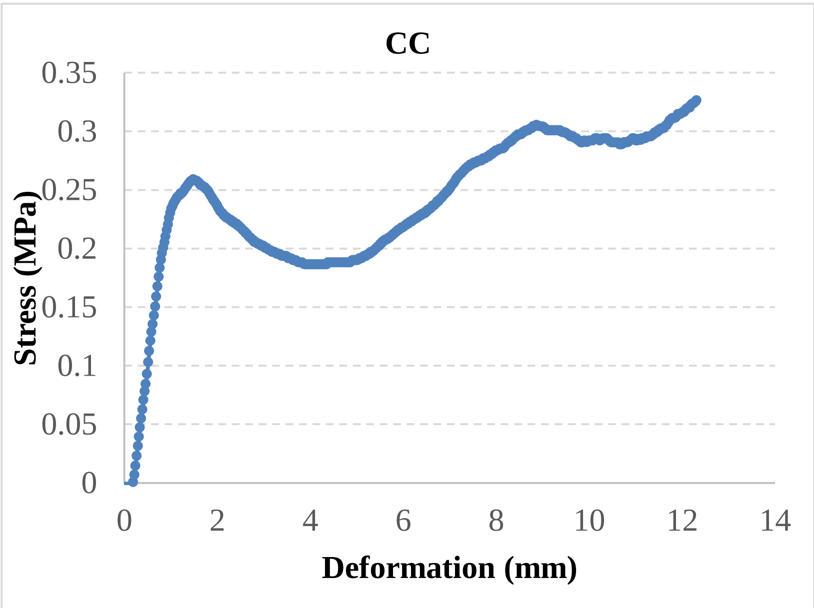
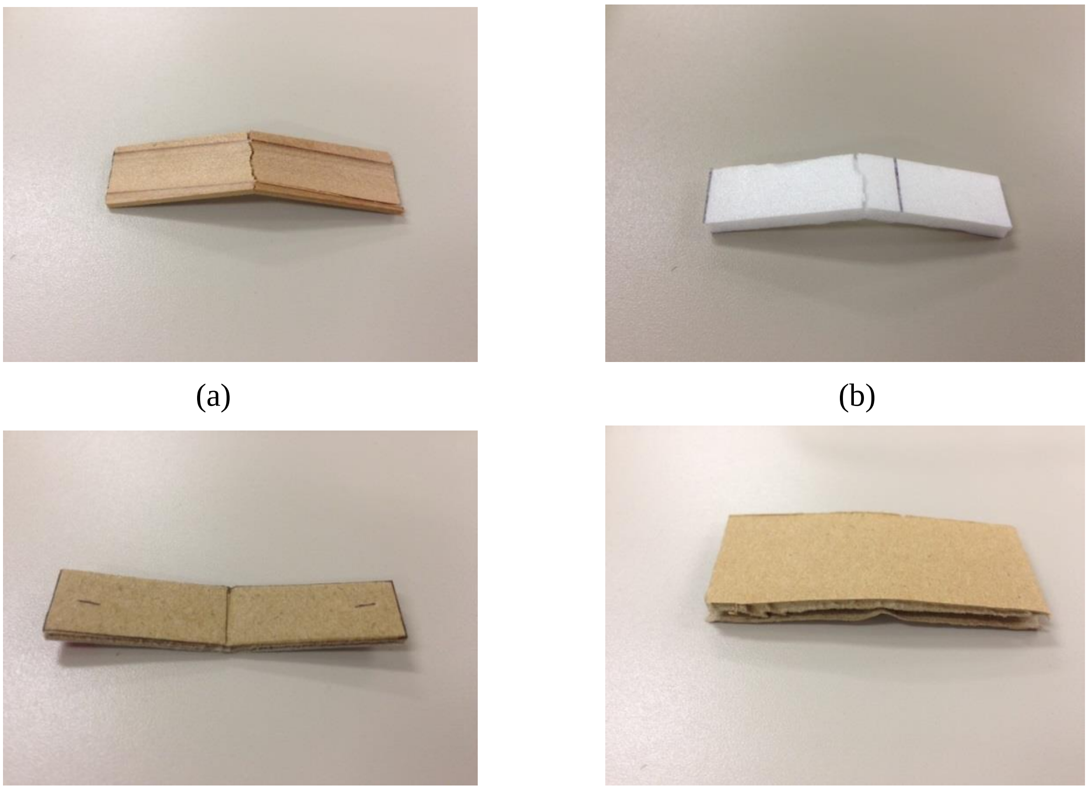


Fig. 5-4 Stress-deformation curve of CC in bending test.

The tested simples for CW and PT, it could be seen obvious cracks occurred approximately at the middle of the specimens. In the opposite, there was not any cracks happened in PB and CC which included particular properties of paper pulp products.



(a) (b)

(c) (d)

Fig. 5-5 Fractured appearance of four specimen samples

(a: CW, b: PT, c: PB, d: CC)

Maximum stress values were shown in figure 5-6. Because PB and CC were not ruptured even maximum stress has been reached, maximum stress could not be defined as a rupture stress value for PB and CC. The resulting maximum stress for the four simples are clear that the bending stress of 61.74MPa was occurred in CW which was strongest strength compared to the other three simples. Minimum stress of CC was 0.259MPa and PT has a maximum stress of 1.606MPa. In addition, although mechanical property of PB was similar to CC, the maximum stress of PB was 6.423MPa. The significant different maximum stress between CW and the other three simples indicated that natural cellulosic material was stronger than the chemical compound materials.

For the strength property of bio-board, which is similar to CW also has a large stress for bending test. Compared with the other three PT, PB and CC, bio-board with a range of 34.52MPa ~ 41.25MPa (calculated in chapter 4) bending stress was much larger. Therefore, bio-board could also have potential applying in packaging materials.

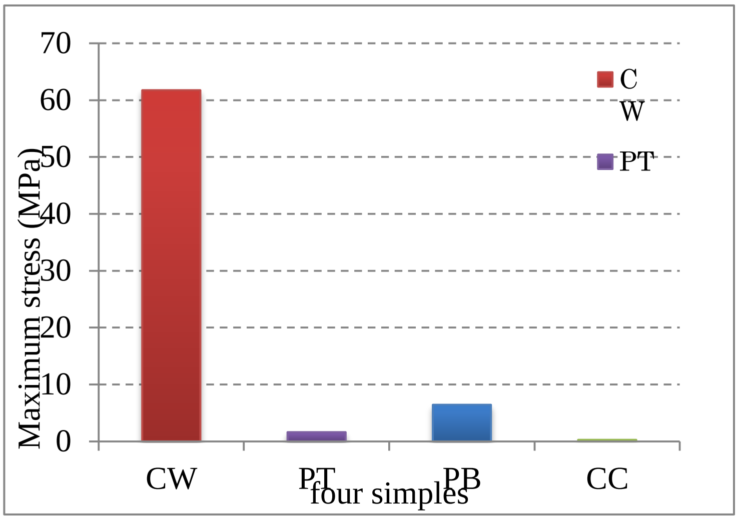


Fig. 5-6 Maximum stress of four simples.

5.4 Strength comparison with wheat straw bio-board

Agricultural residues are known as not only corn straw, also cereal straws. Among them, wheat straw has big potential because of its wide availability and low cost (Sarkar, 2012; Kim, 2004). Additionally, wheat straw has a large production in the world whose chemical property is similar with corn straw. The average yield of wheat straw is 1.3−1.4 kg/kg of wheat grain, with a world production of wheat estimated to be around 680 million tons in 2011. Wheat straw contains 35−45% cellulose, 20−30% hemicelluloses, and around 15% lignin, which makes it an attractive bio-board raw material and other value-added products (Jose, 2012).

In this chapter, strength property of wheat straw is mainly studied comparing with the strength of corn straw. Because wheat straw and corn straw has many commonalities, also they are classified as biomass material. If the commonality of mechanical property between wheat straw and corn straw could be found, it would help researchers to find more similar biomass raw materials to produce bio-board. In addition, the waste which has big potential to be reused is going to be recycled for the limited natural resource is dying day by day.

Generally, board making process was applied using wheat straw. The flow of board making process was the same as corn straw bio-board making process. Besides, pretreatment was prepared using the same devices. Then, strength test including both bending test and tensile strength test was conducted. Finally, rupture stress comparison was made to investigate the difference in the strength of bio-board.

5.5 Materials and methods

5.5.1 **Board making experiment**

Wheat (*Triticum aestivum L*) straws were used in this work according to the NARO (2014). Wheat straw was harvested on June 10^{th}, 2013 at the Mie Prefecture farm. After harvesting, grains were removed. Straw were left in a ventilated storage air-dried for two months.

Five processes applied in previous experiment are used in the present study which are cutting, soaking, grinding, compressing and drying showed in figure 5-7.

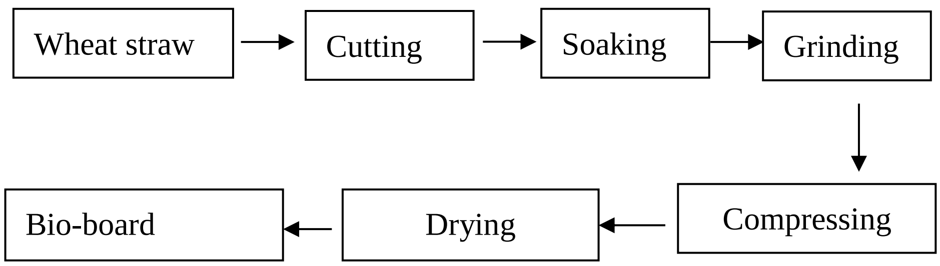


Fig. 5-7 flow chart of board making process

During pretreatment, dry corn straws were cut into chips using an electric cutter, then soaked in water at 22℃ for 168 hours for softening the straw fiber. In soaking process, wheat straw fiber absorbed moisture from water condition. Wheat straw was felt softer than corn straw even though they both were soaked with the same condition.

Soaked wheat straw was then fiberized (pulped) by using an atmospheric refiner with conical blades (Model A Beatfiner. Satomi. Corp.). The motor capacity is 11kw×4p-200, 60Hz, rotational speed is 1750 r.p.m (60Hz). The maximum flux control is 0.05-0.1m3/min. Air pressure is 0.6MPa required. Grinding part is an assembling conical cutter with blades. Dimension of cutter is 2.5mm×3.0mm×8° (blade width × slot width × blade angle).

Fiberization of corn straws at atmospheric pressure was carried out by passing the damp cut straw along with running water through the refiner' rotating blades. During grinding process, fiber bundles would be fiberized by milling. Accordingly, milled-corn straw was sieved to possess particle size using a screen with the hole dimension of 0.5mm×0.5mm.

Four bio-boards were planned to be made with the experimental condition shown in table 5-2. They are named A1, B1 which were made with the pressure of 2MPa and the other two boards named A2, B2 were made by 8MPa

Table 5-2 Experimental condition for making wheat straw bio-board

bio-board No.	pressure (MPa)	dying temp. (℃)
A1,B1	2	110
A2,B2	8	110

5.5.2 **Strength test**

Bending tests and tensile strength tests were conducted for the purpose of analyzing the mechanical properties of wheat straw bio-board. Specimens cut from A1 and A2 were prepared for bending test and specimens from B1, B2 were used for tensile strength test.

First, in the Three-Point Bending Test, 10 specimens were trimmed to 50mm× 20mm × 1.2mm rectangular beam following standard JIS procedures and recommendations (JIS Z2248:1996). All dimensions were measured with an accuracy of + 0.02 mm. Capacity of 100 N load cell was fixed on a motor, applied at a uniform rate of 15mm/min in its vertical direction. Furthermore, the deformation signal was measured by a potentiometer. Both signals of force and deformation were transmitted into an amplifier and A/D convertor then logged in a computer. The bending stress of bio-board was obtained by the quotients of bending moment and section modulus of the specimen. Rupture stress was defined by quotients of maximum bending moment and section modulus of specimen when the specimen was fractured. The classic formula for determining the bending stress is the same to corn straw bio-board bending stress calculation in chapter 3.

Second, tensile strength tests were done under deformation control by using a universal testing machine. Speed of cross-head was 15mm/min. 8 specimens taken from A2 and B2 bio-board named TP1 ~ TP4 were subject to tensile strength tests following standard JIS procedures and recommendations (JIS z 2201).

Normal stress is defined by the quotients of axial load applied on specimen and original cross-sectional area of the specimen. Rupture stress was obtained when the axial load reaches to maximum value while the specimen was fractured. Rupture stress is calculated by the formula mentioned in chapter 3.

5.6 Results and discussion

5.6.1 **Board making**

Four bio-boards were made by the board making process proposed in this study. It was successful for making bio-board using wheat straw. The dimension of bio-boards were 100 mm×100 mm in area and in the range of 2.0mm-2.2 mm in thickness figure 5-8 shows the image of wheat straw bio-board. From the surface and the bottom of bio-board, it showed that the color of two sides is brown and it is darker than corn straw. Compared to corn straw board, few individual big fibers could be seen on both sides in wheat straw board. It implied that wheat straw was not refined uniform well. However the section was smooth with approximate the same height. Thus, dimension of bio-board was not affected by non-uniform fiber.

a. surface b. bottom

c. section

Fig. 5-8 Wheat straw bio-board

5.6.2 **Strength comparison**

Strength comparison for wheat straw (WS) and corn straw (CS) was discussed as follows. Ten specimens were applied in bending stress test, unfortunately, one specimen from A1 board was accidentally failed, it is why only four specimens were displayed in figure 5-9. Generally, bending test resulted that in board A1 stress value increased with the increase of deflection until reaching a maximum value, however the peak of the curve is not obvious. Second, the curves of stress-deflection almost approach a straight line before the fractures occurred. Specimens fractured at approximately 1.5mm~2mm of deflection. Bending stress is called rupture stress when the specimen fractured. After specimen fractured, bending stress decreased to zero, however, bending stress still appears a few because some fibers were still remained connecting. The trend of bending stress-deflection curve varied proportional increase with the same behavior as CS bio-board.

In figure 5-10 displayed the results of bending test from B1 board. The trend of stress-deformation curve showed the same behavior with A1, however, fractures were faster than A1 for the specimens fractured before 2mm deformation.

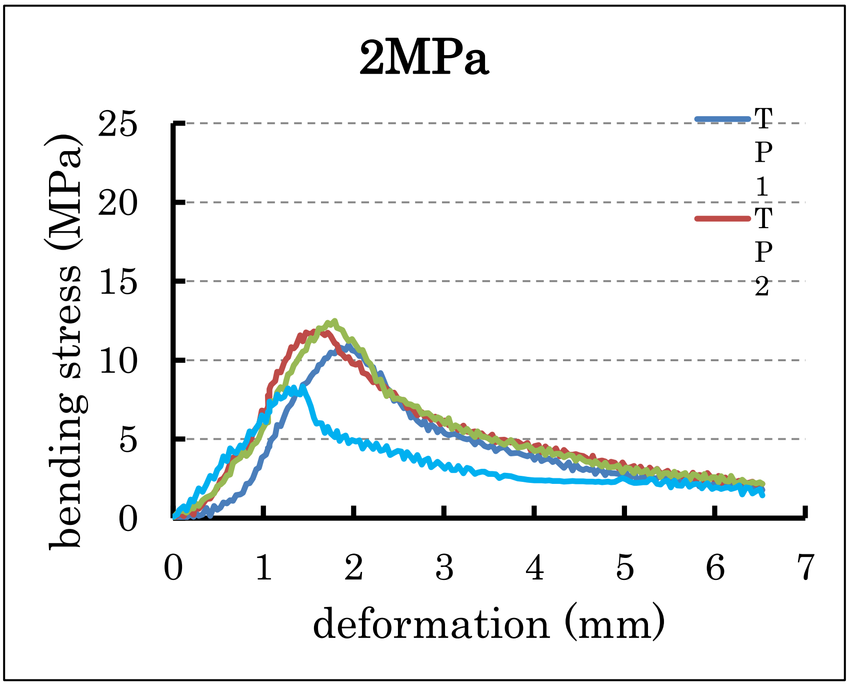


Fig. 5-9 Stress-deformation curve of A1 WS bio-board in bending test

At the proportional line part, B1 increased more steeply than A1 which implied that elastic modulus in B1 is greater than A1.

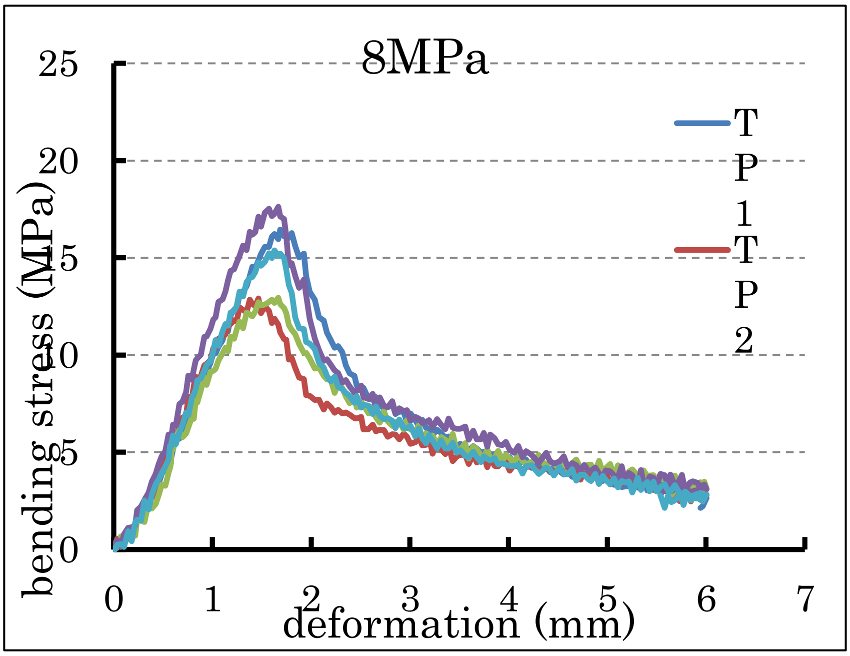


Fig. 5-10 Stress-deformation curve of B1 WS bio-board in bending test

Boards named A2, B2 were used for tensile strength tests and the stress-strain curves are shown in figure 5-11 and figure 5-12. All the curves showed that in the beginning of the stress-strain curve, tensile stress increases as strain becomes large, after the tensile stress reaches the maximum value, specimens fractured, then, tensile stress decreases suddenly to zero. The maximum value is called tensile rupture stress. It could be found that specimens which was from board A2 fractured with a low rupture stress, in the opposite, specimens cut from board B2 showed a high rupture stress value and also the fractured point was at a larger strain value than the one in board A2.

The trend of stress-strain curve received from WS bio-board showed similar behavior to the one of CS bio-board. It could be considered that first, WS and CS are cereal residues which had almost the same chemical compound. Second, fibers contained in the both raw materials shows similar length of range which is from (0.5mm-2.9mm) for CS and (0.4mm-3.2mm) for WS (Mudit.C, 1998). Therefore, board making process is feasible for WS to produce bio-board and the WS board appears certain strength for application.

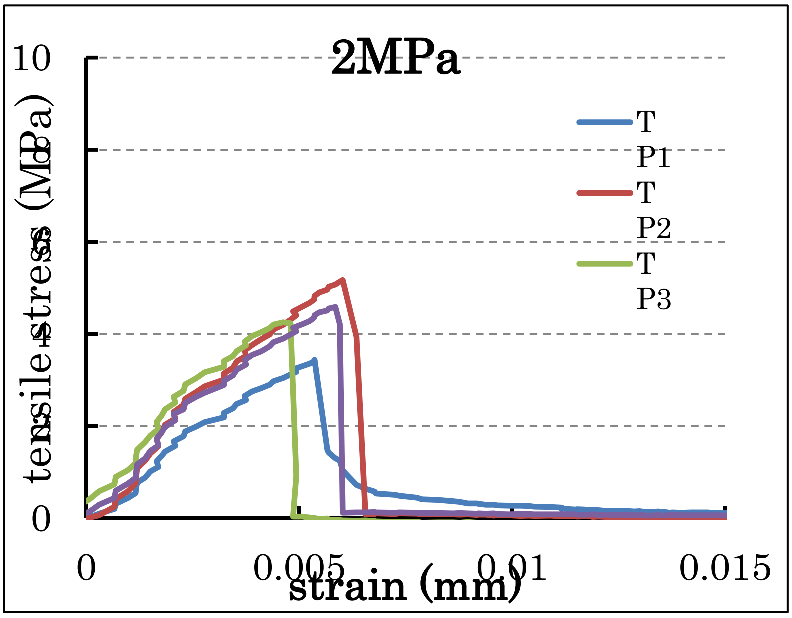


Fig. 5-11 Stress-strain curve of A2 WS bio-board in tensile strength test

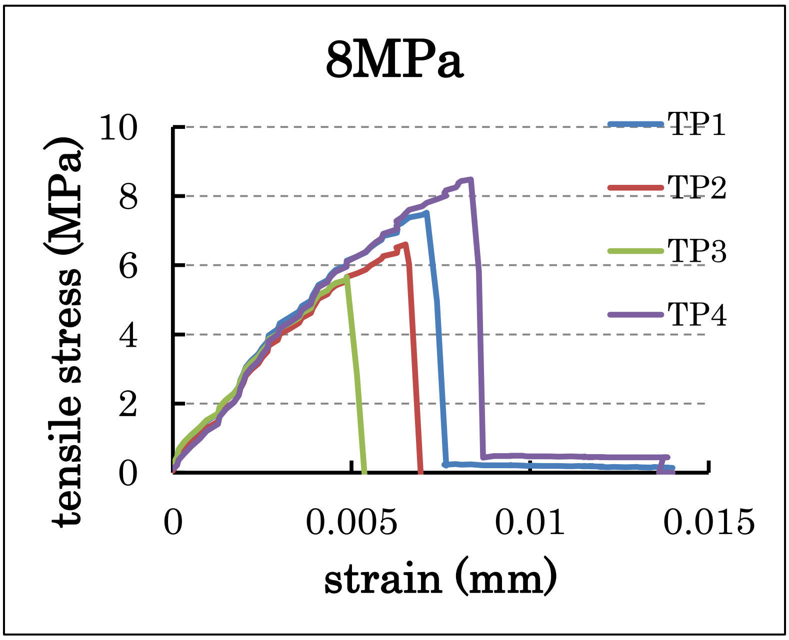


Fig. 5-12 Stress-strain curve of B2 WS bio-board in tensile strength test

In the comparison of rupture stress (RS) for WS and CS, the results were displayed in figure 5-13. Under the condition of applied pressure 2MPa, it can be seen that RS increased with the increase of applied pressure. For bending test, the RS of WS was obviously smaller than the RS of CS. Furthermore, compared to RS of CS the tensile strength RS of WS varied similarly to the former resulting in bending test. Generally, variation of rupture stress for WS board resembles CS board, however, the strength CS showed stronger than WS. It may be explained by some reasons such as the length of fiber in CS was shorter than WS and the content of cellulose was different and so on.

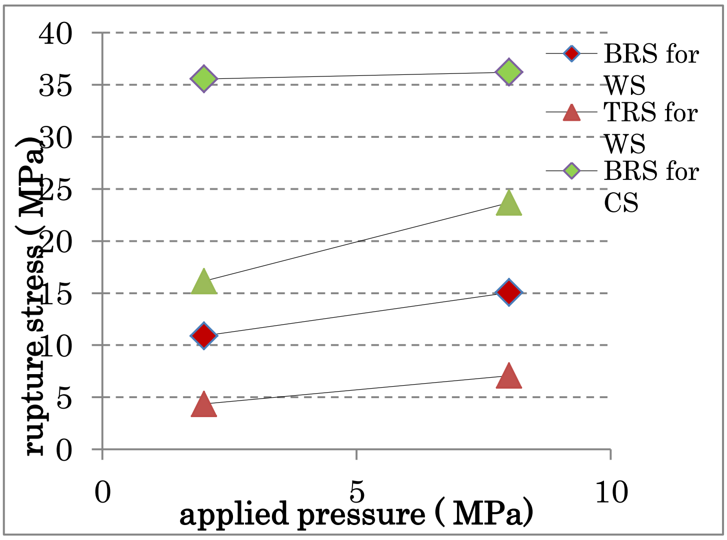


Fig. 5-13 Rupture stress comparison for WS and CS

5.7 Conclusion

After the bending stress investigation of CW, PT, PB, CC, resulting conclusions could be known as follows.

1) The variation stress-deformation for CW was mostly similar to bio-board, on the other hand, PT, PB and CC showed peaks and valleys in stress-deformation curves.
2) Cracks only occurred in CW and PT after specimen fractured, however, PB and CC were yield without any cracks.
3) Resulting maximum stress of the four simples for bending test indicated that strength of CW was the strongest.
4) Compared with the other three PT, PB and CC, bio-board with a range of 34.52MPa ~ 41.25MPa bending stress was much larger.
5) Investigation of bio-board mechanical property was made in this chapter using wheat straw as a raw material. The resulting conclusions could be known as follows:
6) It was successful for making bio-board using wheat straw. The process for the board making is feasible.
7) The trend of bending stress-deflection curve varied proportional increase with the same behavior as CS bio-board before fracture. In tensile strength test, variation of stress-strain curve for WS board was also similar to CS bio-board.
8) Rupture stress of strength test, variation of rupture stress for WS board resembles CS board, however, the strength CS showed stronger than WS.

Therefore, it could be also potentially applied in packaging materials. Another advantage of bio-board is that chemical adhesive was not added in board making process, thus, low cost and friendly environmental characteristic is a representation of bio-board comparing to other chemical compound package materials.

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