# Development of Particleboard Made from Inner Part of Oil Palm Trunk Utilizing the Chemical Components of Raw Materials as an Adhesive

**RAHMA NUR KOMARIAH**

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## Contents

# Chapter I

## *General Introduction*

### 1.1. Development of agro-based particleboard

Particleboard is one of wood-based composite that manufactured from lignocellulosic materials (usually wood), primarily in the form of particles, combined with synthetic resin adhesives and bonded together by hot pressing. In recent years, the demand for wood-based composites has increased dramatically around the world (Sellers, 2001; Youngquist, 1999; Zheng *et. al*., 2007). As the global forest area continues to decline (FAO & UNEP 2020), there are legitimate concerns about diminishing wood resources. This declining forest might affect the supply of raw materials for wood-based composite products. The utilization of non-wood lignocellulosic resources such as agricultural residue will undoubtedly play a substantial role in the future. The quantities of agricultural wastes are increasing due to increase in agricultural activities, with most produced commodities (2000-2019) are cereals, sugar crops, sugar cane and oil crops (FAO, 2019). Such agricultural wastes have remained largely under-utilized owing to lack of the technology for their effective utilization. At present, a great deal of attention has been given to the conversion of agricultural wastes into value added products

(Akaranta & Wankasi, 1998). Based on the both problems of forest preservation and effective utilization of agricultural wastes, the production of particleboard using agricultural wastes is expected to become important in the future.

Many researchers give most attention to developed particleboard using agricultural wastes, e.g., wheat-cereal straws (Mo *et. al.*, 2001; Wang & Sun, 2002; Cheng *et. al.*, 2004; Sain & Phantapulakkal, 2006), rice husks (Leiva *et. al*., 2007), vine (Ntalos & Grigoiou, 2002), kenaf (Xu *et. al*., 2005; Kalaycroglu & Nemli, 2006), bamboo (Hiziroglu *et. al*., 2005; Sudin & Swamy, 2006), bagasse (Widyorini *et. al*., 2005; Liao *et. al*., 2016), sunflower stalks (Nemli, 2003), cotton carpel (Alma *et. al*., 2005), sorghum bagasse (Iswanto *et. al*., 2014; Khazaeian *et. al*., 2015; Kusumah *et. al*., 2016), and oil palm (Khalil *et. al*., 2007; Hashim *et. al.* 2011a; Lamaming *et. al.* 2013).

**Table 1.1.** Chemical characteristics of various agricultural-based lignocellulosic materials (Rowell *et. al*., 2000; Ramaswamy & Tschirner, 2005)

| Fiber Source | Cellulose (%) | Hemi-celluloses (%) | Pectin (%) | Lignin (%) | Water Extractive (%) | Fat-Waxes (%) | Ash (%) |
|---|---|---|---|---|---|---|---|
| Cotton | 82.7 | 5.7 | - | 0 | 1 | 0.6 | 1.2 |
| Hemp | 68.1 | 15.1 | 0.8 | 10.6 | 2.1 | 0.7 | 2.5 |
| Flax | 78.5 | 9.2 | 0.3 | 8.5 | 1.8 | 0.2 | 1.5 |
| Jute | 64.4 | 12 | 0.2 | 11.8 | 1.1 | 0.5 | - |
| Ramie | 68.8 | 13.1 | 1.9 | 0.6 | 5.5 | 0.3 | - |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Kenaf | 55-59 | 18-20 | 4.5-5 | 6.8-8 | - | 0.6-1.8 | - |
| Manila | 63.2 | 19.6 | 0.5 | 5.1 | 1.4 | 0.2 | - |
| Miscanthus | 40 | - | 31 | 20 | 6 | - | - |
| Palm tree | 52.7 | - | 21.6 | 15.1 | 7.2 | - | - |
| Sisal | 65.8 | 12 | 0.8 | 9.9 | 1.2 | 0.3 | - |
| Banana | 60-65 | 6-8 | - | 5-10 | - | - | 4.7 |
| Residue tea | 23.2 | 30.2 | - | 32.14 | 32 | - | - |
| Hazelnut shell | 34.5 | 20.6 | - | 35.1 | - | 18.2 | 8.2 |
| Rice Straw | 26-36 | 23-28 | - | 12-14 | - | - | 14-20 |
| Coffee husk | 19-26 | 24-45 | - | 18-30 | - | - | 6-7 |
| Coffee hull | 40-49 | 25-32 | - | 33-35 | - | - | 0.5-1 |
| Wheat straw | 33-38 | 26-32 | - | 17-19 | - | - | 6-8 |
| Coniferous (softwood) | 40-45 | 7-14 | - | 26-34 | | - | 1 |
| Decduous (hardwood) | 38-49 | 19-26 | - | 23-30 | | - | 1 |

Table 1.1 shows the chemical properties of some common agricultural-based lignocellulosic materials. Comparing to wood, agricultural-based lignocellulosic materials are usually characterized by a lower lignin content and a higher hemicelluloses

content. These materials seem to be suitable for the production of wood-based materials such as particleboard, especially, binderless boards that take advantage of the self-bonding by hemicellulose degradation.

### 1.2. Current situation of oil palm tree plantation

FAO (2019) reported that there is approximately 28.2 million ha of oil palm plantations in the world. Indonesia is the top country with total area of oil palm plantations was approximately 14.3 million ha (Directorate General of Estate Crops, 2019). While, global annual production of oil palm biomass is estimated around 214.5 million tons (Tye *et al*. 2016). As an agricultural plant, the oil palm tree (*Elaeis guineensis*) has become a major contributing crop in Indonesia's growing economy. Oil contributes to only 10% of the total biomass produced by the oil palm tree, and once the oil palm tree reaches 25 years old, its oil productivity generally diminishes, and the oil palm tree should be replanted. Estimated that approximately 34 million tons/year of felled trunk were produced (Ismail & Mamat, 2002). The palm oil industry usually generates a large amount of waste. The waste includes the fruit, kernel shell, mesocarp, and fronds of palm trees as well as the oil palm trunk (OPT) (Hambali & Rivai 2017). And estimated 10% of oil palm waste had been reused while the leftover became a residue and contributed to severe

environmental problems (Dungani *et al*., 2016). OPTs are generally underutilized and usually burned because incineration is a fast and cheap method for plantation area clearing. Such practices cause environmental problems like air pollution. In contrast, OPT waste can be left on the plantation field to decompose and become natural fertilizer. However, this activity is poorly managed and impractical for large amounts of waste.

### 1.3. Characteristics of oil palm trunk

Oil palm trunks are classified as monocotyledonous plants consisting of vascular bundles (VB) and parenchyma tissue. Vascular bundles consist of fiber bundles, metaxylem or vessels, protoxylem, protophloem and axial parenchyma (Bakar *et. al.*, 2008). Ahmad *et. al.* (2010) stated that the diameter of the OPT fiber bundle ranged from 0.3-0.6 mm. The fiber bundle walls in OPT are thicker than in parenchyma tissue. Therefore, the VB component is a strength parameter of OPT, especially VB with old cells (Darwis *et. al.*, 2013). The VB portion is increases towards the outside of the stem and vice versa for parenchyma tissue. The mechanical properties of OPT is enhanced with the increasing number of VB (Iswanto *et. al.*, 2010), so that the mechanical properties on the outer part (1/3 diameter of OPT) of the OPT are superior to the inner part (2/3 diameter of OPT). Meanwhile, the network parenchyma consists of parenchyma cells that contain

sugar and starch, work as energy and food storage tissues (Bakar *et. al.,* 2008). The anatomical structure of this OPT is very porous so it is easy to absorb and release water (Choowang & Hiziroglu, 2015). This affects the dimensions of the OPT.

In addition, the chemical characteristics of OPT have also been investigated by several previous studies. Husin *et. al.* (1985) has studied the chemical properties of OPT and found that the OPT has lignin and lignocelluloses contents markedly lower but shows higher content of extractive, as well as water and alkali soluble than coconut wood and rubberwood. In a separate study, Mansor and Ahmad (1990) observed that the lignin content was evenly distributed throughout the tree except that the inner part in the upper region was slightly deficient in the component whilst the bottom contained an excessive amount. The lignin content range varies between 15% and 21.7%. The result is consistent with the fact that the number of fibrous VB increases towards the outer part and thickening of the older VB gives rise to the higher lignin content of the lower trunk. The ash content also observed to be similar throughout the trunk with the range varies between 3.0% and 3.3%.

Rahim *et. al.* (1987) was studied on the chemical components of OPT, such as free sugars and starch. It was found that freshly felled OPT may yield up to 10% free sugars and 25% starch. In advance, Mansor and Ahmad (1990) reported that a total

content of free sugars of 2 to 10% throughout the trunk height. The inner part was found to elaborate higher proportion of free sugars as shown by the methanol-water extractive whilst the outer part had the lowest. According to analysis by high performance liquid chromatography (HPLC) revealed sucrose, glucose, and fructose as the three main free sugars of the oil palm trunk. Further the authors indicated that analysis free sugar by acid hydrolysis of oil palm trunk produced higher number of sugars, ranging between 48% and 70%. Examination of the HPLC trace of the acid hydrolyzate showed the present of six sugar components namely glucose, xylose, galactose, arabinose, mannose, and rhamnose, with glucose being the major component (35 to 48%) followed by xylose (11 to 16%). Based on standard TAPPI of chemical analysis, Bakar *et. al.* (1998), using OPT of Tenera variety, had also concluded that a gradual decrease in lignin content and cellulose from outer part to inner part, whilst starch content was increased. Ash and silica contents were found high value at the inner part. The soluble analyses using hot water, cold water, alcohol benzene and NaOH 1% were also found high proportions at the inner part.

### 1.4. Potential of oil palm trunk resources for wood-based composites

Oil palm trunk (*Elaeis guineensis* Jacq.) is a non-wood lignocellulosic material that has the potential to be used as an alternative raw material for wood-based products. Researches on composite products from OPT have only used the outside of the OPT while the inside of the OPT is still rarely used. This is due to differences in the characteristics of the outside and inside of the OPT.

In line with point 1.3 that explained about the characteristics of OPT, Bakar *et. al*. (2008) stated that only the outside of the OPT can be used as building construction materials, the inside of the oil palm trunk can only be used as animal feed because it contains a lot of starch. Iswanto *et. al.* (2010) also stated that the outer (1/3 of diameter the OPT) can be used as raw material for furniture or light construction materials. This is influenced by the VB portion of the OPT which is higher on the outside (Rahayu, 2001). However, the utilization of 2/3 diameter of the OPT (the inner part) needs to be optimized to increase the added value of OPT. One way of optimizing OPT is with the technology of modifying the inner part of the OPT on various products such as composite products.

Research into wood-based composite products using oil palm trunk (OPT) can be classified mainly into two categories. One category is research using synthetic resin adhesives. Phenol formaldehyde resin and urea formaldehyde resin adhesives have been

used for various wood-based composites such as plywood and particleboard (Baskaran *et. al.* 2019; Hartono *et. al.* 2017; Hashim *et. al.* 2010). It is generally known that the physical and mechanical properties of wood-based composites are not sufficient. One reason for this is that water-soluble components including saccharides inhibit the curing of adhesives (Bockel *et. al.,* 2019; Jumhuri *et. al.,* 2014). To resolve this problem, an increase in resin content and the utilization of other high-performance adhesives such as pMDI (polymeric diphenylmethane diisocyanate) were proposed (Hermanto & Massijaya, 2018; Prabuningrum *et. al.,* 2020).

The other category is research into binderless boards that do not use synthetic resin adhesives. In this case, chemical components in raw material are denatured by heat and moisture to develop adhesiveness (Hashim *et. al.,* 2011a, 2011b; Lamaming *et. al.,* 2013; Lamaming *et. al.,* 2014). However, the performance of binderless boards is generally very poor when compared with boards that use a suitable synthetic resin adhesive, especially one that is water-resistant.

Generally, conventional synthetic resin adhesives are composed of assorted chemical substances derived from fossil resources. However, their use will be inescapably restricted in the future due to decreases in the stock of fossil resources also the requirement of human health. Considering the backdrop, a high-performance board

fabricated by a method that does not use synthetic resin adhesive is desired. Recently, ammonium dihydrogen phosphate and sucrose has been observed as wood-based natural adhesives in manufacturing of wood-based particleboards (Umemura *et. al.*, 2017; Zhao *et. al*., 2018) and bamboo-based particleboard (Widyorini, 2020).

### 1.5. Characteristics of ammonium dihydrogen phosphate (ADP) and sucrose as an adhesive

Ammonium dihydrogen phosphate (ADP) belongs to a large family of compounds having general chemical formula: $MH_2XO_4$, where M = K, Rb, Cs, $NH_4$; or the mixed component of such elements H = H or D; X = P or As (Xue & Ratajczak, 2005). Studies on ADP crystal attract interest in view of their dielectric, antiferroelectric and optical properties and their varied use as electro-optic modulators, second harmonic and parametric generators (Cook & Smith, 1974). ADP is used as fire-prevention agent for fabric, timber and paper; as well as fire-prevention coating, and dry powder for fire extinguisher. For food grade it is mainly used as a fermentation agent, nourishment, and so on. It is also used as a high effective non-chloride nitrogen and phosphorous compound fertilizer in agriculture.

Among the sugars found in nature, sucrose plays a dominant role in food processing and agriculture. It is naturally present in many plants, in varying quantities. In some plants, it serves as the storage product, although, in other plants, the storage product comprises different sugars such as fructose (fruit), or complex carbohydrates such as starch (corn, potatoes) (Huberlant, 2003). Sucrose is a disaccharide which is composed of the monosaccharide glucose and fructose link via and ether bond between C1 on the glucosyl sub-unit and C2 on the fructosyl unit (Tondi *et. al.*, 2012). There are many plants from which sucrose commercially extracted, including the date palm, sorghum, sugar maple, and the two most important worldwide sources: sugar cane, and sugar beet. Sucrose is used as a sweetener in both the food and pharmaceutical industries (Rowe *et. al.*, 2003).

Sucrose is extremely soluble in water; hence it has high mobility in water and its abundance in hydroxyl groups (Bock & Lemieux, 1982). Moreover, sucrose is chemically labile and can undergo hydrolysis in the presence of and acid in solution to form fructose and glucose (Kwok *et. al.*, 2010). In addition, sugar modifies its structure when the temperature rises up to 160 °C (Queneau *et. al.*, 2007). During this process, called caramelization, some isomerization of sucrose is happening and highly reactive furanic derivatives are produced (Taher & Cates, 1974). At present researches, sucrose was tried

to use as an adhesive in the manufacturing of wood-based composites (Umemura *et. al.*, 2013, 2014; Widyorini *et. al.*, 2016; Liao *et. al.*, 2016; Zhao *et. al.*, 2014; Lamaming *et. al.*, 2013). Moreover, with the addition of ADP and heating, sucrose could be change into a high-water-resistance substance (Umemura *et. al*., 2017) and used as an adhesive for manufacturing particleboard (Zhao *et.al*., 2018, 2019a; Widyorini, 2020). The 5-hydroxymethyl-2-furfural (5-HMF) is considered one of the most important sucrose-derived products responsible for the bonding mechanism of sucrose-ADP-based adhesive. Zhao *et.al* (2019a) stated that the main reactions during the curing process were the dehydration condensation of furan compounds and the Maillard reaction. The resulting substance is mainly polyfuran and linked by C-O-C and C=N-C linkages. The possible mechanism of the sucrose-ADP adhesive system proposed by Zhao *et. al*. (2019a) is shown in Fig. 1.1.

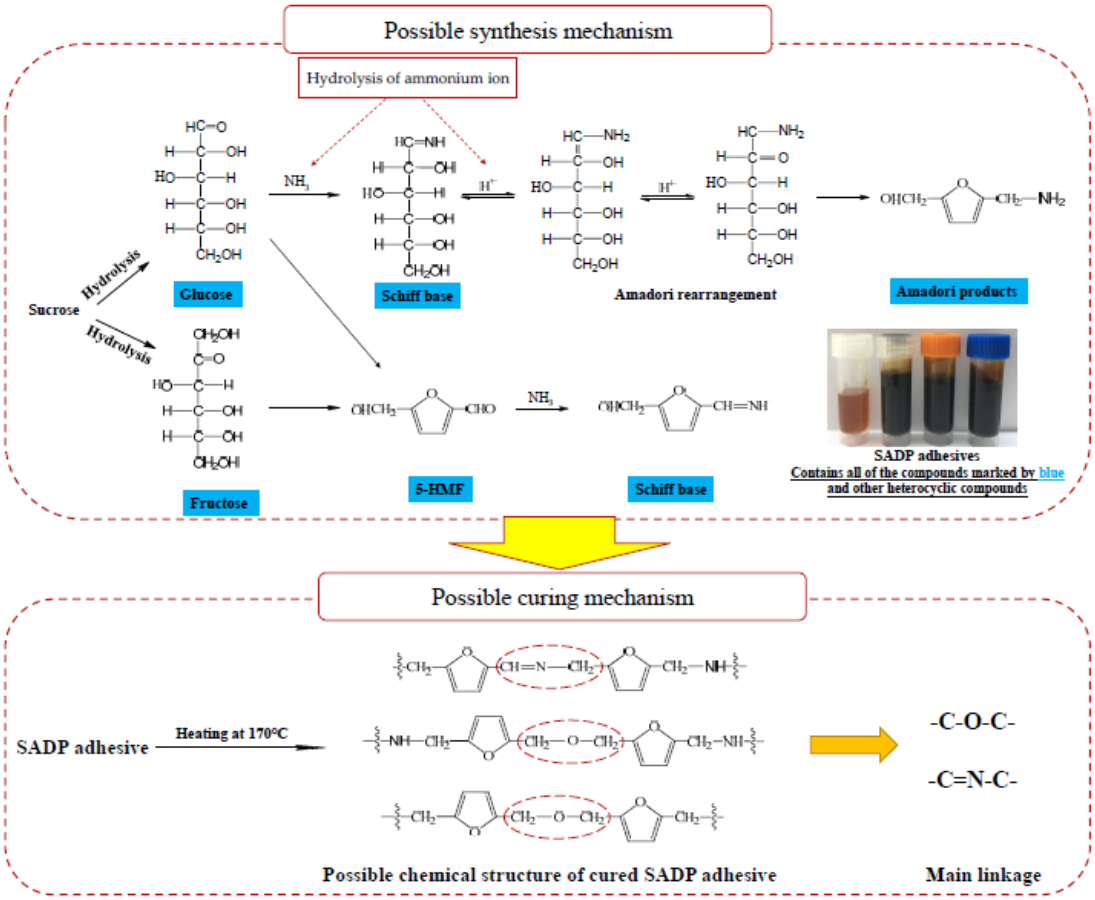



**Fig. 1.1.** The possible mechanism of sucrose-ADP adhesive (Zhao *et.al*. 2019a).

## 1.6. Objectives

The oil palm trunk is agricultural waste, but has potential as lignocellulosic material. Studies on manufacturing particleboards using oil palm trunk have been conducted in recent years. However, the physical and mechanical properties of particleboards were usually insufficient. Therefore, the objective of this study is to develop particleboard with good physical and mechanical properties using inner part of OPT by methods that do not depend on fossil resources as much as possible. Specifically,

particleboards were manufactured by utilization of chemical components of inner part of OPT, ADP and sucrose.

The present thesis consists of four chapters;

1. Chapter I comprises literature reviews on the subject covering the development of agro-based particleboard, current situation of oil palm plantation, characteristic of lignocellulosic materials from oil palm trunk, and characteristic of ammonium dihydrogen phosphate (ADP) and sucrose as an adhesive.
2. Chapter II describes on the chemical composition the inner part of OPT and the binderless particleboards were manufactured based on the results of the thermal analyses of OPT added ADP. The effects of ADP as an additive on the properties of binderless particleboard then investigated. Moreover, the bonding mechanism underlying the production of the binderless particleboards was investigated using Fourier Transform Infrared (FTIR) spectroscopy.
3. Chapter III demonstrates the ADP's influences on the physical and chemical changes of a water-soluble extractive of the inner part of OPT. The changes of extractive into insoluble substances were observed and the formation of furan compounds derived from sugars of extractive was analyzed using FTIR. The

effect of the water-soluble extractive on binderless particleboard with the addition of ADP was also investigated.

4. Chapter IV deals with the effect of adding sucrose with ADP as an adhesive on the improvement of physical and mechanical properties of particleboard using the inner part of OPT. The effects of different ratios of sucrose and ADP and different resin contents were investigated. In addition, the termite and decay resistance and the infrared spectra (IR) of the particleboard were examined.

# Chapter 2

## *Development of binderless particleboard using the inner part of oil palm trunk and ammonium dihydrogen phosphate*

### 2.1. Introduction

Commonly, the inner part of oil palm trunk becomes waste due to its low dimensional stability, low strength, and low durability, whereas the outer part of OPT could potentially be used for light-weight building materials and furniture (Bakar *et. al.*, 2008; Erwinsyah, 2008). Some research groups have attempted to use the inner part of OPT by applying treatments involving resin impregnation and densification (Khalil *et. al.*, 2012; Hartono *et. al.*, 2016). These treatments usually require the use of synthetic resins derived from fossil resources. Considering the future of fossil resources and the disadvantageous of synthetic resins, the manufacturing of wood-based products without the use of synthetic resins is desired. For example, the method used to manufacture binderless particleboard is attractive because it does not need synthetic resins. Research on the manufacture of binderless particleboard from mixtures of the outer and inner parts of OPT (i.e., the entire OPT) has been conducted (Hashim *et. al.*, 2010, 2011a; Lamaming *et. al.*, 2013, 2014), however many problems — especially poor water resistance — have

been encountered. The poor water resistance would be due to the chemical components of OPT that containing rich of sugar, starch, and saccharides in the parenchyma. Mansor and Ahmad (1990) clarified that the main free sugar in the OPT was sucrose and glucose.

As reviewed in Chapter 1, Umemura *et. al.* (2017) found that sucrose could be changed into a high-water-resistance substance by the addition of ammonium dihydrogen phosphate (ADP) and heating. Usually, ADP is used as a food additive, a flame retardant, a pharmaceutical agent, and a fertilizer ingredient. Zhao *et. al.* (2018) reported that a mixture of sucrose and ADP could be used as a high-water-resistance adhesive for manufacturing particleboard.

Considering the results of the Umemura *et. al.* and Zhao *et. al.*, also Mansor and Ahmad studies, the use of ADP may be effective for the manufacture of binderless particleboard that uses only the inner part of OPT. Therefore, in this chapter, investigating the effects of ADP as an additive on the properties of binderless particleboard using the inner part of OPT was conducted. The chemical composition and thermal properties of the inner part of OPT were observed. The bonding mechanism underlying the production of the particleboards was investigated using Fourier Transform Infrared spectroscopy (FTIR). In addition, color changes on binderless particleboard and the effects of ADP on the water-soluble components of OPT were observed.

## 2.2. Materials and methods

### 2.2.1. Preparation of materials

Oil palm trunk (*Elaeis guineensis* Jacq.) with the approximate age of 30 years and an approx. 60-cm diameter was obtained from Bogor, Indonesia. After the oil palm tree was cut down, the inner part of the trunk (the inner two-thirds of the diameter) was cut and chopped into chips. The chips were dried under sunlight and then oven-dried at 70°C for 12 h to reach a moisture content between 1 and 3%. The dried chips were ground to particles with a ring flaker (Pallman Maschinenfabrik, Zweibrucken, Germany).

The particles were screened with a sieving mill (Iida sieve shaker, Iida Seisakusho, Yokohama, Japan), and the particles that remained between the aperture sizes of 2 and 0.25 mm were used as the raw material. Extra-pure-grade ammonium dihydrogen phosphate (ADP) was purchased from Nacalai Tesque (Kyoto, Japan) and used without further purification. The ADP was dissolved in distilled water at the concentration of 20 wt%. The pH and viscosity of the solution at 21°C were 3.9 and 3 mPa•s, respectively.

### 2.2.2. Chemical composition analyses

For the chemical composition analyses, OPT particles were pulverized (passed 150 μm) with a Tissue Lyser II (Qiagen, Tokyo) at 25 Hz for 3 min. The OPT powder (200 mg) was sequentially extracted twenty times with methanol at 60°C, then extracted six times with hexane at room temperature, extracted five times with distilled water at

60°C, and then freeze-dried. The supernatants from each extraction were accumulated as total extractive; hereafter the freeze-dried powder was referred as the cell-wall residue (CWR) that was used for lignin, ash measurement, and polysaccharides analyses. The lignin content was determined based on the Klason method (TAPPI T 222 om-02, 2002). The ash content was determined by incinerating CWR at 800°C for 17 h (Yamamura *et. al.*, 2013).

The matrix polysaccharides were hydrolyzed with 2 M trifluoroacetic acid (TFA) at 100°C for 5 h. The resulting monomeric sugars that were further calculated as the hemicellulose content were derivatized by the alditol acetate method (Hayashi, 1989) and quantified by a gas chromatography-mass spectrometry (GC-MS) analysis (GCMS-QP 2010 Plus, Shimadzu, Kyoto, Japan) (Miyamoto *et. al.*, 2018). Myo-inositol (Nacalai Tesque, Kyoto, Japan) was used as an internal standard for quantification. In parallel, pellets left after the TFA hydrolysis were treated with Updegraff reagent (Updegraff, 1969), washed with distilled water and acetone, and then completely hydrolyzed with $H_2SO_4$ (Hattori *et. al.*, 2012). The released glucose was quantified using a Glucose CII test kit (Wako Pure Chemicals Industries, Osaka, Japan). The percentage of cellulose was then calculated.

The starch analysis was performed by amylase treatment (Yamamura *et. al.*, 2013).

Approximately 1 g of OPT powder was used as sample. Then 80% (v/v) ethanol was added to the samples also a solution of thermostable α-amylase (Megazyme International Ireland, Wicklow, Ireland) that had been diluted 30-fold with 100 mM sodium acetate buffer (pH 5.0, 540 µl). Furthermore, amyloglucosidase (Megazyme International Ireland, Wicklow, Ireland) was added to the tubes. The mixture was incubated in a rotary reactor (Heatblock Rotator SN-48BN, Nissin Rika, Saitama, Japan) at 5 rpm and 50°C for 30 min. The tubes were centrifuged (10,000×*g*, 2 min), and the supernatants were collected to measure the amount of liberated glucose. The glucose absorbance was calculated as the starch content.

The free sugar was measured by 80% (v/v) ethanol at 40°C for 16 h (Trebbi & McGrath, 2004). The sucrose assay kit SCA20-1KIT, 20 assays (Sigma-Aldrich, St. Louis, MO), a fructose assay kit (C/F) (Biovision, Palo Alto, CA) and Glucose CII test kit (Wako Pure Chemicals, Osaka, Japan) were applied to the extracted solution. Spectrophotometry (UV-VIS Spectrophotometer VIS-2600, Shimadzu) was performed at a wavelength of 505 nm. Glucose was used as a standard for creating the calibration curve.

### 2.2.3. Thermal analyses of OPT

OPT particles were pulverized into powder (passed 150 μm). The 20 wt% ADP solution was poured into the OPT powder at 0–40 wt% solid content based on the weight of the OPT powder. The mixtures were freeze-dried for 48 h, and ADP-added OPT samples were obtained. A thermogravimetric analysis (TGA) was carried out using the TGA 2050 thermogravimetric analyzer (TA Instruments, Tokyo). The ADP-added OPT samples were scanned from room temperature to 400°C at an increased rate of 5°C/min under nitrogen purging with a flow rate of 70 mL/min.

A differential scanning calorimetry (DSC) also carried out with a DSC 2910 differential scanning calorimeter (TA Instruments, Tokyo). Prior to preparing the sample, a small hole was made in the cover of the aluminum pan using a tweezer. Each ADP-added OPT sample for the DSC analysis was encapsulated in an aluminum pan and was scanned from room temperature to 400°C under nitrogen purging with the flow rate of 50 mL/min and an increased rate of 5°C/min.

### 2.2.4. Manufacturing of particleboards

The 20 wt% ADP solution was sprayed onto OPT particles with ADP solid contents from 2 to 40 wt% based on the OPT particles weight. The sprayed particles were

dried at 90°C for 24 h to reach a moisture content <5%. The dried particles were mat-formed using a 300 mm × 300 mm forming box. The mat was hot-pressed at 180°C for 10 min with a 5-mm distance bar to control the board thickness. The particleboard with 0 wt% ADP was obtained from particles that were not sprayed with an ADP solution.

To prevent blisters, after 1 minute of pressing the press pressure was reduced for 30 sec. The size of the particleboard was 300 mm × 300 mm × 5 mm, and the target density was 0.8 g/cm$^3$. The manufactured particleboards were conditioned for 1 week at 20°C and approx. 60% relative humidity before undergoing physical and mechanical tests.

### 2.2.5. Color measurement

The surface color of the particleboards after conditioning was measured by a spectrophotometer (CM-2600d, Konica Minolta, Tokyo) under the light source of D65 and UV with an observer angle of 10° and specular component included. The sensor head of the spectrophotometer was 8 mm in dia. The CIELAB color parameters on the variable's lightness ($L^*$) and the chromaticity coordinates on the green-red axis ($a^*$) and blue-yellow axis ($b^*$) were measured. These data were used to calculate the color difference ($\Delta E^*$) according to the following formula (JIS [Japanese Industrial Standards] Z 8729, 2004):

$$\Delta L^* = L^* - L_0^*;\ \Delta a^* = a^* - a_0^*;\ \Delta b^* = b^* - b_0^* \quad (2\text{-}1)$$

$$\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (2\text{-}2)$$

Where $L_0^*$, $a_0^*$, and $b_0^*$ are the particleboard with 0 wt% ADP. Nine locations on each surface of the particleboard were measured, and the average value and standard deviation based on 18 locations were calculated.

### 2.2.6. Evaluation of the particleboards

The particleboards were tested according to Japanese Industrial Standards (JIS A 5908, 2003). The bending properties of the particleboards; the modulus of rupture (MOR) and the modulus of elasticity (MOE) were calculated based on the static three-point bending test. The test was carried out on a 200 mm × 30 mm × 5 mm sample with the effective span was 150 mm, and the cross-head speed was 10 mm/min. The internal bond (IB) test was conducted with a tension loading speed of approx. 2 mm/min on a 50 mm × 50 mm × 5 mm specimen.

The thickness swelling (TS) after water immersion at 20°C for 24 h was measured on a 50 mm × 50 mm × 5 mm specimen. The water absorption (WA) of the specimens and the pH values of the soaked water were measured. After the TS test, the specimens were subjected to a cyclic aging treatment: drying at 105°C for 10 h, hot water immersion

at 70°C for 24 h, drying at 105°C for 10 h, immersion in boiling water for 4 h, and drying at 105°C for 10 h. The changes in thickness and weight of the specimens throughout the treatments were determined. The means values and standard deviation were calculated from five replications of each test. The values of MOR, MOE, and IB of the boards were corrected for each target density based on the regression lines between the obtained values and the specimen densities.

### 2.2.7. Statistical analysis

Data for each test were statistically analyzed. The analysis of variance was used to evaluate the significance in difference between factors and levels. Comparison of the means was done by using Tukey's HSD post hoc test to identify the significance of differences of each other group at the 95% confidence level. The standard deviation were also calculated from the data and are shown as errors bars in each corresponding figure.

### 2.2.8. Fourier transform infrared spectroscopy (FTIR) measurement

Fourier transform infrared spectroscopy (FTIR) analysis was performed using specimens after the bending test and specimens after the cyclic aging treatment. Each specimen was ground into powder (passed 150 μm) using a blender (Vita-mix Absolute

3 ABS-W, Osaka Chemicals, Japan). The powder obtained was vacuum-dried at 60°C for 15 h. Infrared (IR) spectra were obtained with an FTIR spectrophotometer (FT/IR-4200, Jasco, Tokyo) using the KBr disk method, and the spectra were recorded by an average of 32 scans at a resolution of 4 $cm^{-1}$ in the 400-4000 $cm^{-1}$ range.

## 2.3.Results and discussion

### 2.3.1. The chemical composition of the OPT inner part

**Table 2.1.** Chemical composition of the inner part of oil palm trunk.

| Chemical composition | Content (% dry weight particle) |
|---|---|
| Cellulose | 30.22 (0.35) |
| Hemicellulose | 14.38 (0.26) |
| Lignin | 20.87 (2.15) |
| Extractive | 26.79 (1.86) |
| Ash | 1.84 (0.002) |

*value in parentheses are standard deviation

The chemical composition of the OPTs' inner part is summarized in Table 2.1 The cellulose and hemicellulose contents were 30.22% and 14.38%, respectively, which were slightly lower than the reported values (Saka *et. al.*, 2008; Hashim *et. al.*, 2011b; Lamaming *et. al.*, 2014) in studies that used the entire OPT. Since the inner OPT consists mostly of parenchyma, the lower value of cellulose agreed with Abe *et. al.* (2013), who

reported that the cellulose content of the OPT parenchyma was four times lower than that of the cellulose in the vascular bundles. The lignin and ash contents that determined herein were similar to those obtained in other studies (Saka *et. al.*, 2008; Hashim *et. al.*, 2011b; Lamaming *et. al.*, 2014).

The extractive content in this study was relatively higher than those reported in the studies of the entire OPT (Saka *et. al.*, 2008; Hashim *et. al.*, 2011b; Lamaming *et. al.*, 2014). According to Bakar *et. al.* (2013), the parenchyma might contribute to the higher extractive content in the OPT. Lange and Simatupang (1994) reported that the extractive of OPT consisted of sugars, amino acids, phenolics, soluble lignin, and water-soluble polysaccharide.

**Table 2.2.** Starch and sugar content of the inner part of oil palm trunk.

| Starch (% dry weight particle) | Free sugar composition on ethanol extraction (% dry weight particle) | | |
|---|---|---|---|
| | Sucrose | Glucose | Fructose |
| 4.45 (0.05) | 5.44 (0.82) | 1.41 (0.11) | 0.99 (0.08) |

*Value in parentheses is standard deviation

In this study, the starch and sugar compounds of the inner part of OPT are shown in Table 2.2. The starch content was approx. 4%, and the sucrose, glucose, and fructose free sugar contents were 5.44%, 1.41%, and 0.99%, respectively. Yamada *et. al.* (2010),

Eze and Ogan (1988), and Mansor and Ahmad (1990) stated that sucrose, glucose, and fructose were the major sugar components in OPT.

### 2.3.2. Thermal analyses

The thermal analyses were carried out to investigate the effect of ADP addition on the thermal properties of the inner part of OPT. Figure 2.1 provides the DSC curves of the OPT particle and OPT with added ADP. The DSC curve for the OPT particle (0 wt% ADP) had one endothermic peak at around 70°C, and two broad exothermic peaks at approx. 270 and 330°C. The addition of ADP brought some changes in the DSC curves. The exothermic peaks at ~270 and 330°C decreased, and a sharp exothermic peak at ~270°C appeared and shifted to ~250°C. An endothermic peak gradually appeared at ~190°C and tended to become sharp with increasing ADP content. An endothermic peak at ~70°C was observed regardless of the ADP content.

Figure 2.2 shows the thermogravimetric (TG) and derivative TG (DTG) curves. In the TG curves (a), a weight reduction of the OPT particle (0 wt% ADP content) was observed at temperatures between 200 and 330°C. According to the DTG curves (b), the OPT particle (0 wt% ADP) content exhibited a significant weight reduction at ~310°C. The temperature for the weight reduction tended to move to lower temperatures with the

increase in the ADP content. A clear two-step weight reduction appeared with the addition of ADP 5 to 40 wt%. The weight reduction was observed at ~180°C when the ADP content was over 20 wt%.

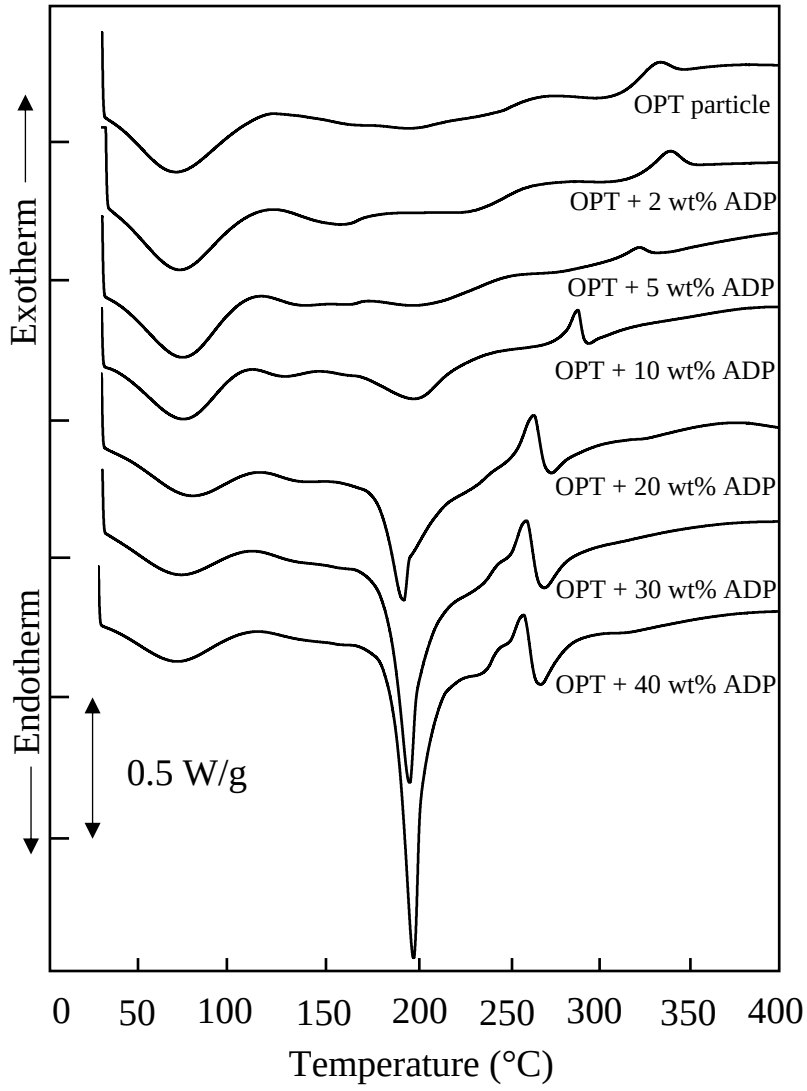



**Fig. 2.1.** DSC of OPT particle and OPT added ADP.

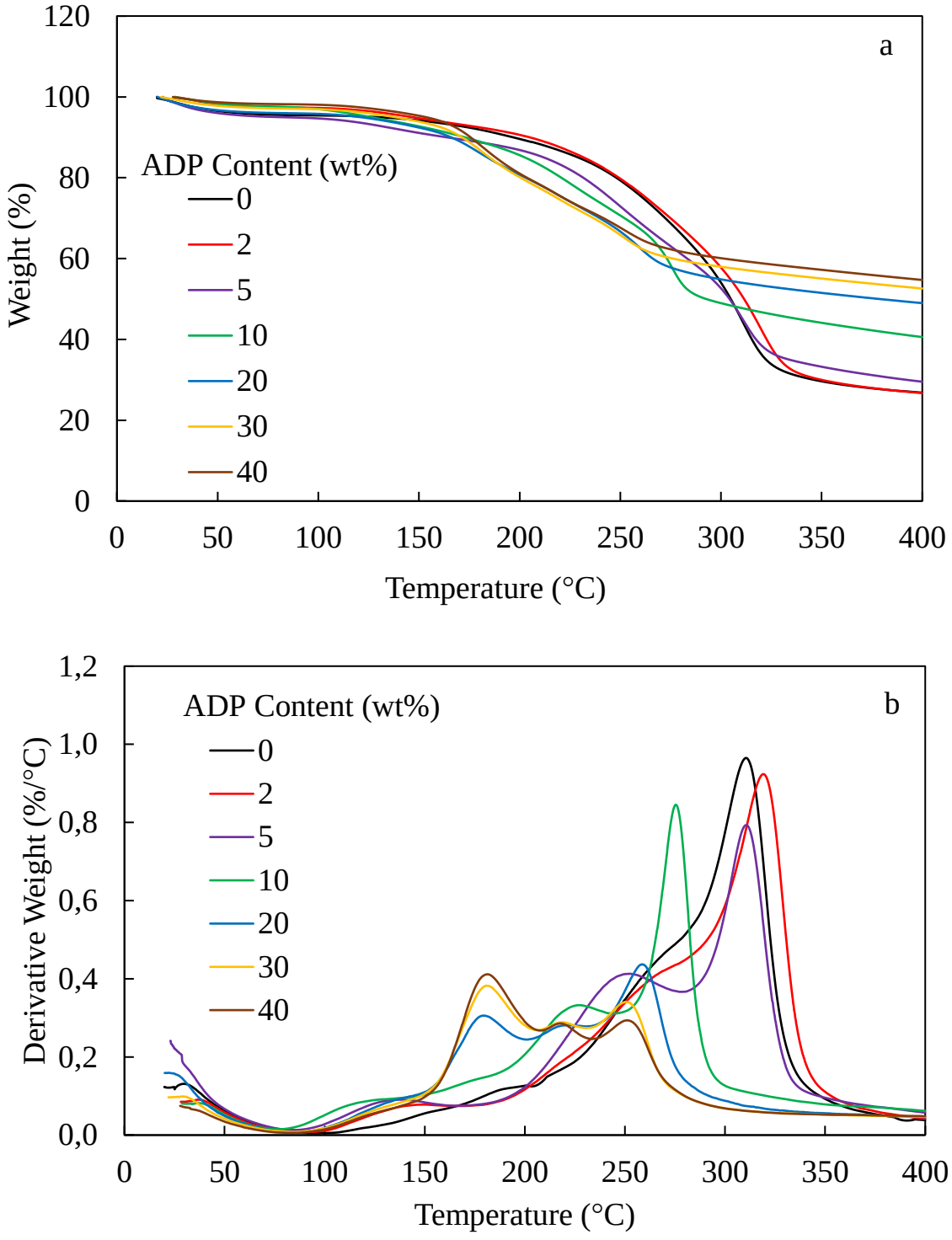



**Fig. 2.2.** Thermogravimetric (TG) (a) and Derivative TG (DTG) (b) of OPT added ADP (wt%).

The thermal properties of OPT with added ADP based on the above-described results were discussed. An endothermic peak of the DSC at ~70°C was observed in all specimens. However, weight reduction was hardly observed in the TG and DTG curves. According to Mehrotra *et. al.* (2010), the effects of the weakening of the hydrogen bonds between carbohydrates was shown as an endothermic peak at ~70°C. Therefore, the endothermic peak at ~70°C of OPT would be due to the weakening of the hydrogen bonds between carbohydrates. The exothermic peak at ~270°C would be attributed to the decomposition of hemicellulose and the slower decomposition of lignin, according to Yang *et. al.* (2007). Shebani *et. al.* (2008) and Sulaiman and Abdullah (2014) suggested that an exothermic peak at ~330°C indicates the decomposition of cellulose. These phenomena were also supported by the weight reduction between 200 and 330°C in TG in this study. When ADP was added to OPT, the degradation of OPT shifted to lower temperatures around 250°C. According to Umemura *et. al.* (2017), ADP showed exothermic and endothermic peaks at ~203°C and 208°C, respectively, and ADP had a weight reduction onset temperature at ~170°C and one-step weight reduction at ~185°C. Abdel-Kadeer *et. al.* (1991) conforming to, that ADP undergoes changes such as melting and phosphoric acid-producing decomposition in this temperature range. The endothermic peaks and marked weight reduction were observed at ~180°C that might be

mainly come from the melting and decomposition of ADP. Thus, the hot press temperature of 180°C was used when manufacturing the particleboard using OPT.

### 2.3.3. Color measurement of the particleboards

Figure 2.3 shows the color of the particleboards on different addition of ADP. The color of the OPT particleboards gradually became dark brown — when observed with naked eyes — with the increase in the ADP content, indicating that marked oxidation occurred due to ADP addition. According to Lai (2001) and Hon and Minemura (2001), the increase in the color change could occur due to the degradation of products from hemicellulose and lignin. Therefore, it was suggested that the OPT particles underwent oxidation easily with the addition of ADP when manufacturing particleboard.

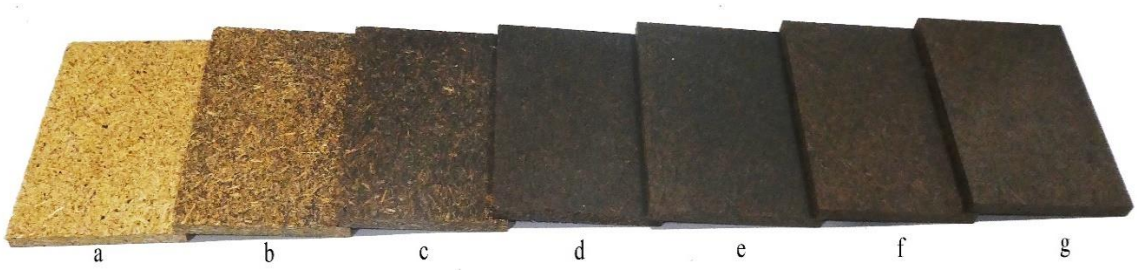



**Fig. 2.3.** The color of the particleboards (a) binderless (0 wt% ADP), (b) 2 wt% ADP, (c) 5 wt% ADP, (d) 10 wt% ADP, (e) 20 wt% ADP, (f) 30 wt% ADP, and (g) 40 wt% ADP.

Figure 2.4 shows the changes in brightness ($L^*$) and color differences ($\Delta E^*$) of the particleboards with different ADP contents. The $L^*$ value of the particleboards gradually decreased with the addition of ADP to 10 wt%; after that, there were no significant changes in the brightness level ($p>0.05$). The $\Delta E^*$ value increased gradually up to 10 wt% ADP content and then decreased slightly until 40 wt% ADP content. Based on the

statistical analyses, the Δ*E** values of the particleboards with the addition of ADP 10 to 40 wt% were not significantly different ($p>0.05$). It can be concluded that the particleboards with the addition of ADP 10 to 40 wt% had similar brightness (*L**) and color differences (Δ*E**).

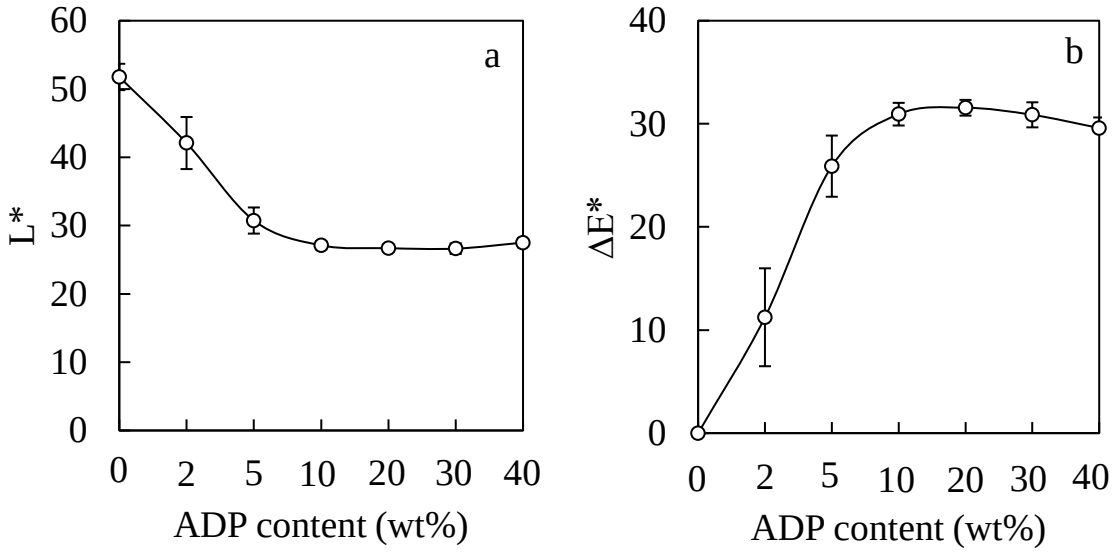



**Fig. 2.4.** Changes in brightness (*L**) (a) and color differences (Δ*E**) (b) of the particleboards.

### 2.3.4. Mechanical properties of the particleboards

Figure 2.5 shows the effect of ADP contents on the bending properties of the particleboards. The MOR and MOE values of the particleboards with 0 wt% ADP content were 5.2 MPa and 1.7 GPa, respectively. These values are similar to the results for a binderless board made from the entire OPT (Lamaming *et. al.*, 2013). The bending properties improved with the addition of ADP. The highest MOR and MOE values were 8.9 MPa and 2.5 GPa, respectively, for the particleboard with 10 wt% ADP. However, the addition of ADP 20 to 40 wt% brought a decline in the bending properties, due to an excess of ADP in the particleboard.

The statistical analyses demonstrated that the MOR values of the particleboards with the addition of ADP 20 to 40 wt% were not significantly different ($p>0.05$) from that of the particleboard with 0 wt% ADP content. These results clarified that the addition of <10 wt% ADP contributed to the improvement of the bending properties of the particleboards.

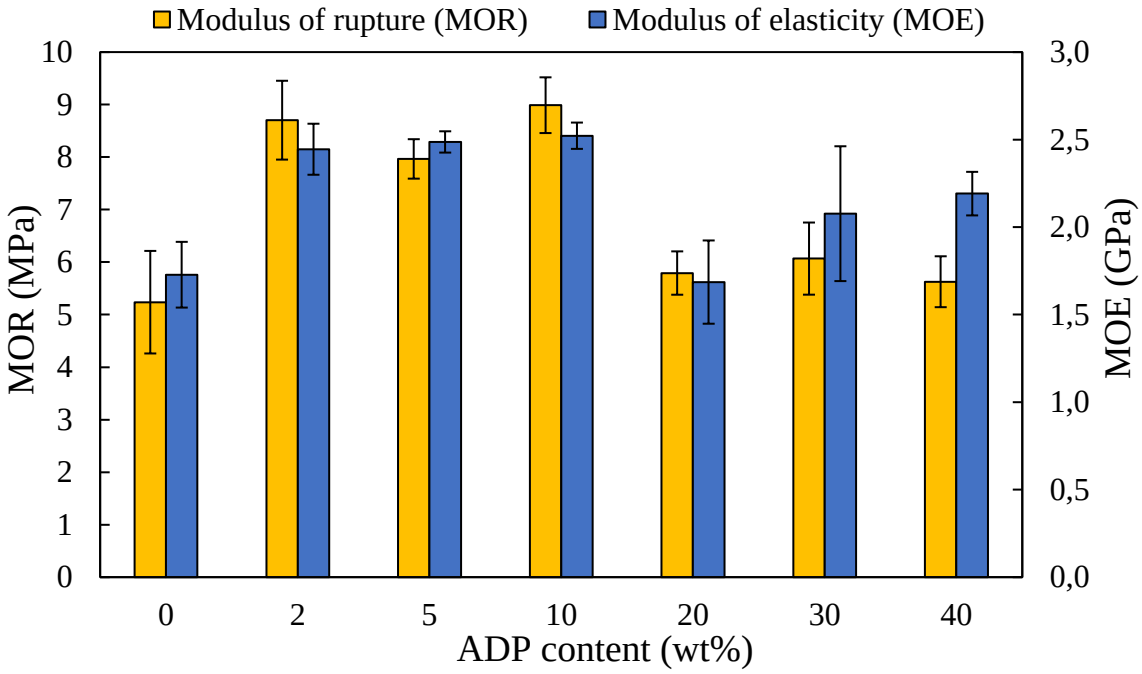



**Fig. 2.5.** Bending properties of the particleboards.

The internal bond (IB) test results (Fig. 2.6) showed a trend that was similar to the bending properties (Fig. 2.5). The IB values of the particleboards with the addition of 2 to 10 wt% ADP were higher than that of the particleboards with 0 wt% ADP. The IB value of the 10 wt% ADP particleboard was the highest (0.53 MPa) and was approx. 64% greater than of that the 0 wt% ADP. The particleboards with 20 to 40 wt% ADP showed IB values that were similar to that of the particleboard with 0 wt% ADP. The addition of <10 wt% ADP provided an improvement in the bond strength between particles.

Judging from the results obtained, the addition of ADP could improve the mechanical properties of particleboards. The optimum ADP content for the mechanical properties of particleboard was 10 wt% in this study.

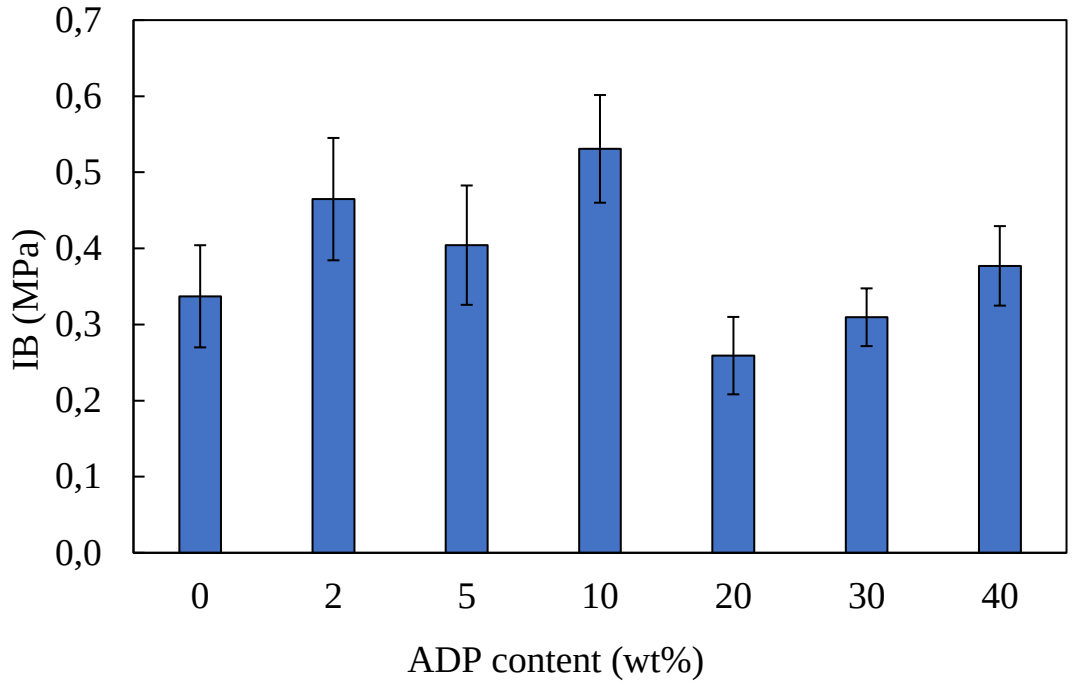



**Fig. 2.6.** Internal bond (IB) of the particleboards.

### 2.3.5. Water resistance of the particleboards

Figure 2.7 shows the effects of ADP content on the TS and WA values of the particleboards. The TS value of the 0 wt% ADP particleboard was the worst (49.7%) compared to the particleboards with ADP addition. The TS value improved gradually with the addition of ADP until 10 wt% and then declined slightly. The best TS value (5.9%) was observed in the particleboard with 10 wt% ADP. Considering the requirement of a TS value <12% in the JIS A 5908 standard, the particleboards with the addition of ADP from 10 to 40 wt% had good dimensional stability.

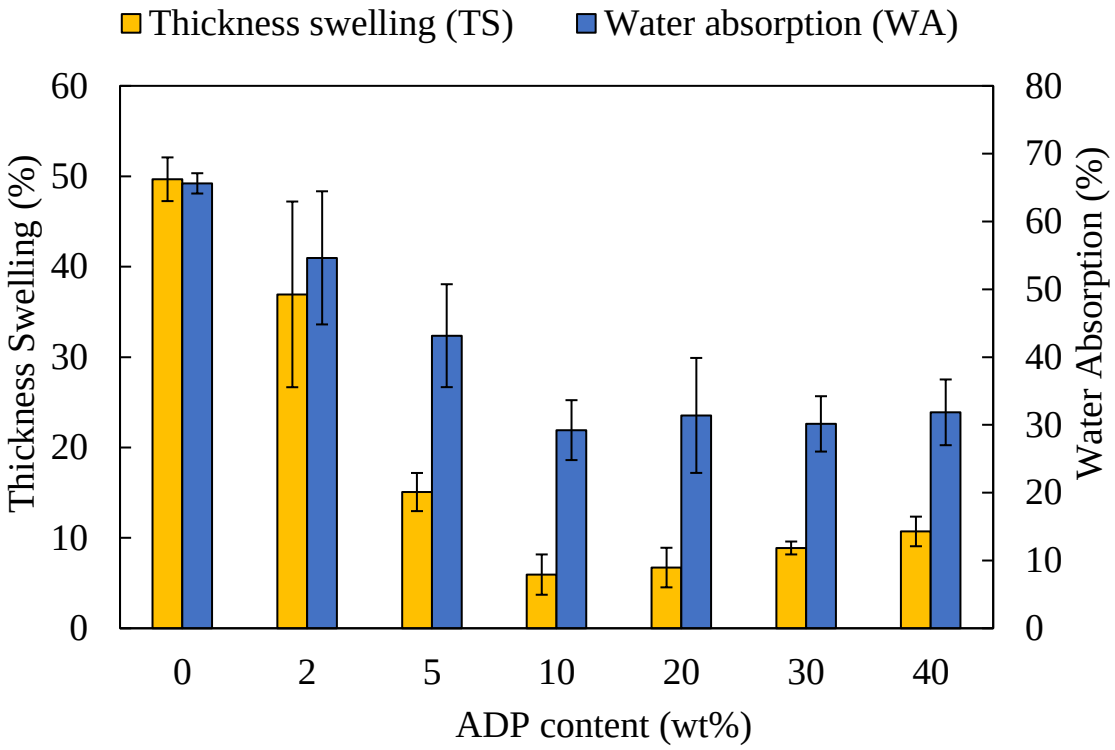



**Fig. 2.7.** Thickness swelling and water absorption of the particleboards.

The WA values showed a similar trend; the particleboard with 0 wt% ADP had the worst WA value (65.6%), and the best WA value was observed in the 10 wt% ADP (29.2%). The pH values of the water solution after the TS test are shown in Figure 2.8. The pH value in the 0 wt% ADP was 4.3, which is similar to that of wood (pH 3.8–6.1) (Hon & Minemura, 2001). The pH values obtained with the addition of ADP ranged from 2.3 to 3.6. The pH value decreased with the addition of ADP until 10 wt% and then increased slightly. This result indicates that the particleboards with added ADP might release some amount of acidic compound from the OPT and ADP, as it is known that ADP melts and decomposes partly at ~170°C, producing phosphoric acid (Abdel-Kadeer *et. al.*, 1991). The increase in the pH value with the addition of 20 to 40 wt% ADP would be due to a large amount of ADP, wherein the pH of ADP was around 3.9.

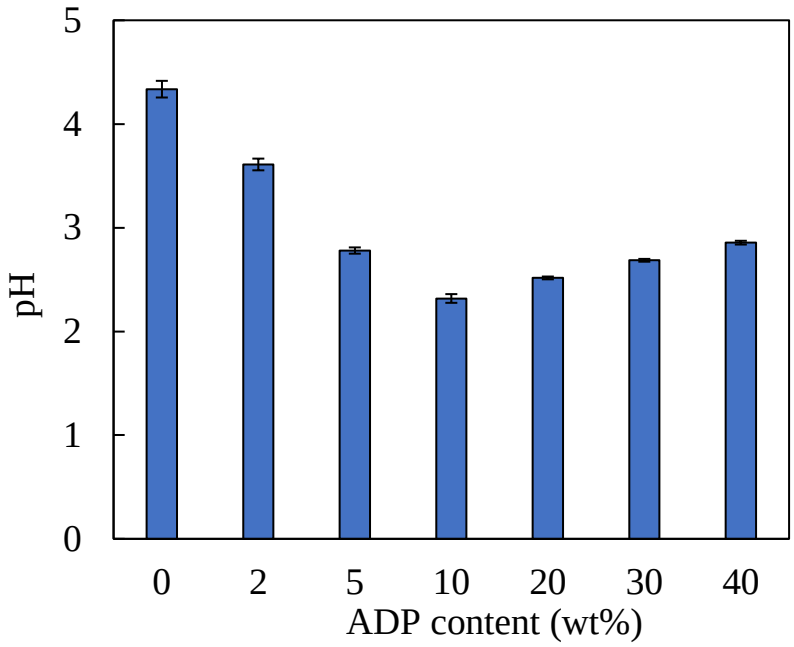



**Fig. 2.8.** pH of soaking water after TS treatment of the particleboards.

To further investigate the water resistance of the particleboards with added ADP, the thickness and weight changes that occurred with cyclic aging treatment were observed. The results are shown in Figure 2.9. In the case of 0 wt% ADP, the thickness change after the water immersion at 70°C was 108%, and the change after the boiling treatment was 132%. At the end of treatment, the final dried thickness change was 87%. Though the particleboard with 0 wt% ADP kept its form even after the treatment, a remarkable increase in the thickness was recognized. However, the thickness changes of the particleboard with 5 wt% or more ADP were greatly suppressed. The final dried thickness change was between 0.2 and 11%, wherein the particleboard with 10 wt% ADP showed the lowest final thickness change (0.2%).

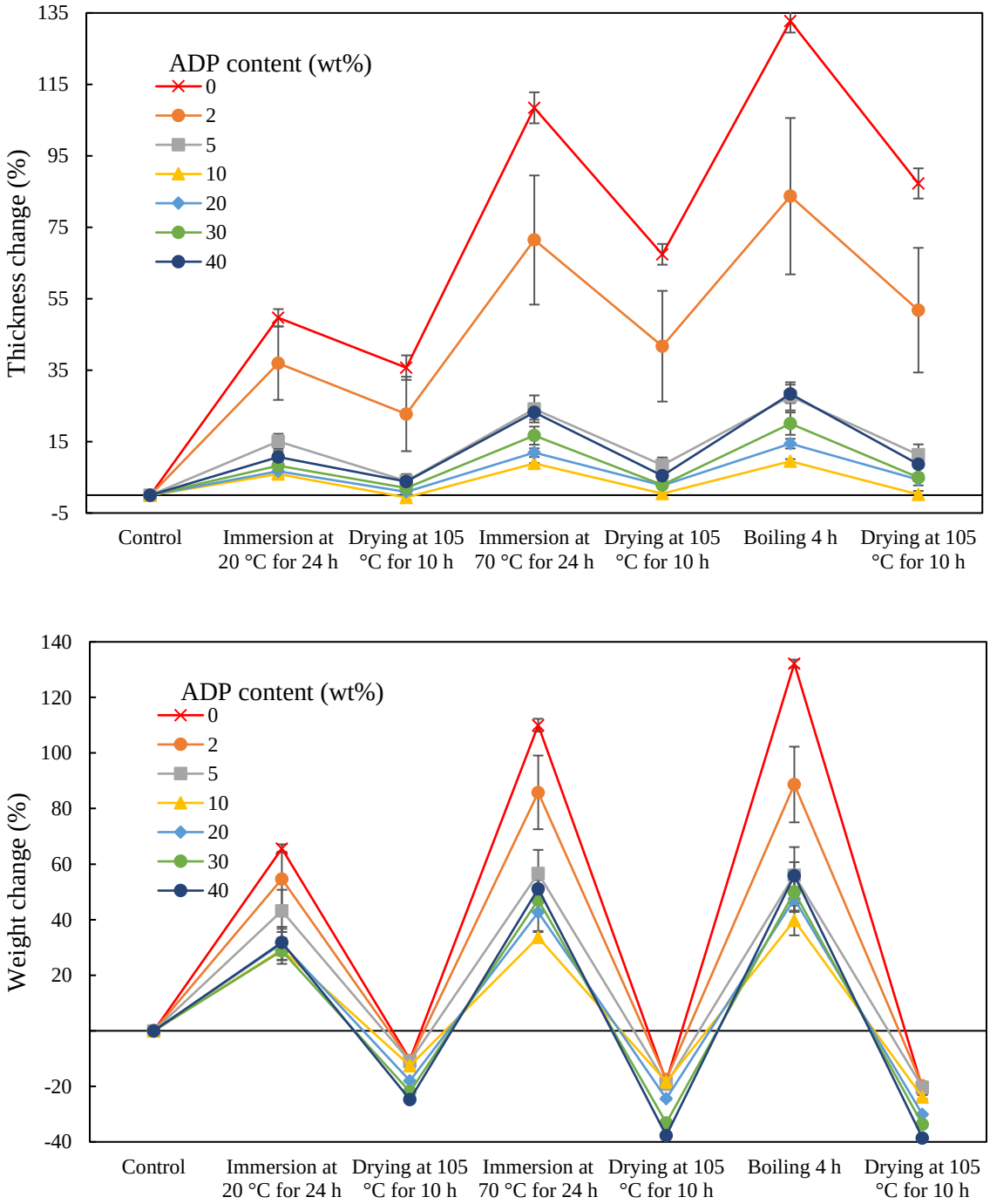



**Fig. 2.9.** Thickness and weight changes of particleboard on the cyclic aging treatment test.

The weight change of the 0 wt% ADP increased in each immersion treatment, and at the last boiling treatment, the weight increased to 132%. After the final drying, the weight change was recorded as −20.64%. This means that elution of water-soluble

components from OPT particles occurred during the treatment. In the particleboards with added ADP, the weight increase was inhibited in each immersion treatment. After the final drying, the weight change was between −23 and −39%. The main cause seemed to be elution of substances related to ADP and some water-soluble components from OPT.

However, the weight decrease remained small despite the portion of added-ADP, which is water-soluble. It was thus clarified that the particleboard with added ADP had good water resistance. In particular, the particleboard with 10 wt% ADP showed excellent water resistance even under harsh treatment.

### 2.3.6. FTIR analysis

The FTIR spectra were measured to clarify the chemical changes of the particleboards due to ADP addition. The noticeable chemical changes of OPT particles and particleboards with the addition of ADP 0, 10, 20, and 40 wt% were recorded on the range of 400-2000 $cm^{-1}$, as shown in Figure 2.10. The peaks at ~1720 and 1635 $cm^{-1}$ (derived from the C=O stretching vibration of conjugated carbonyl compounds) increased slightly with the increase in the ADP content. It is generally known that ADP has absorption peaks at ~1400, 1100, 910 and 530 $cm^{-1}$ wherein the peak at ~1400 $cm^{-1}$ is attributed to the $NH_4$ ion, and the other peaks are attributed to the vibration of $PO_4$ (Abdel-Kadeer *et. al.*, 1991; Umemura *et. al.*, 2017). These peaks gradually increased with the increase in the ADP content. This means that the added ADP had remained in the particleboards.

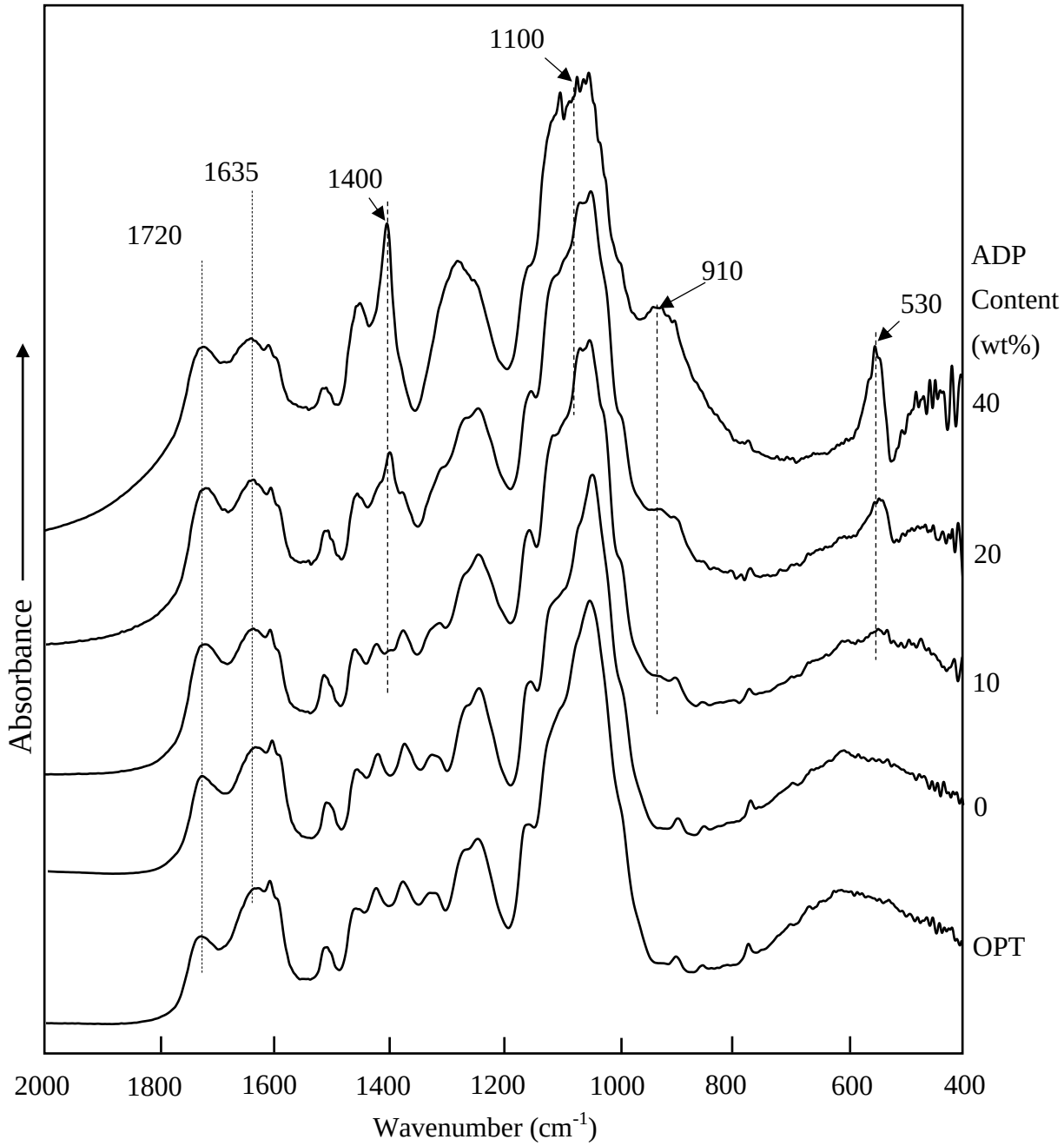



**Fig. 2.10.** FTIR spectra of OPT particle and boards on different ADP content (wt%).

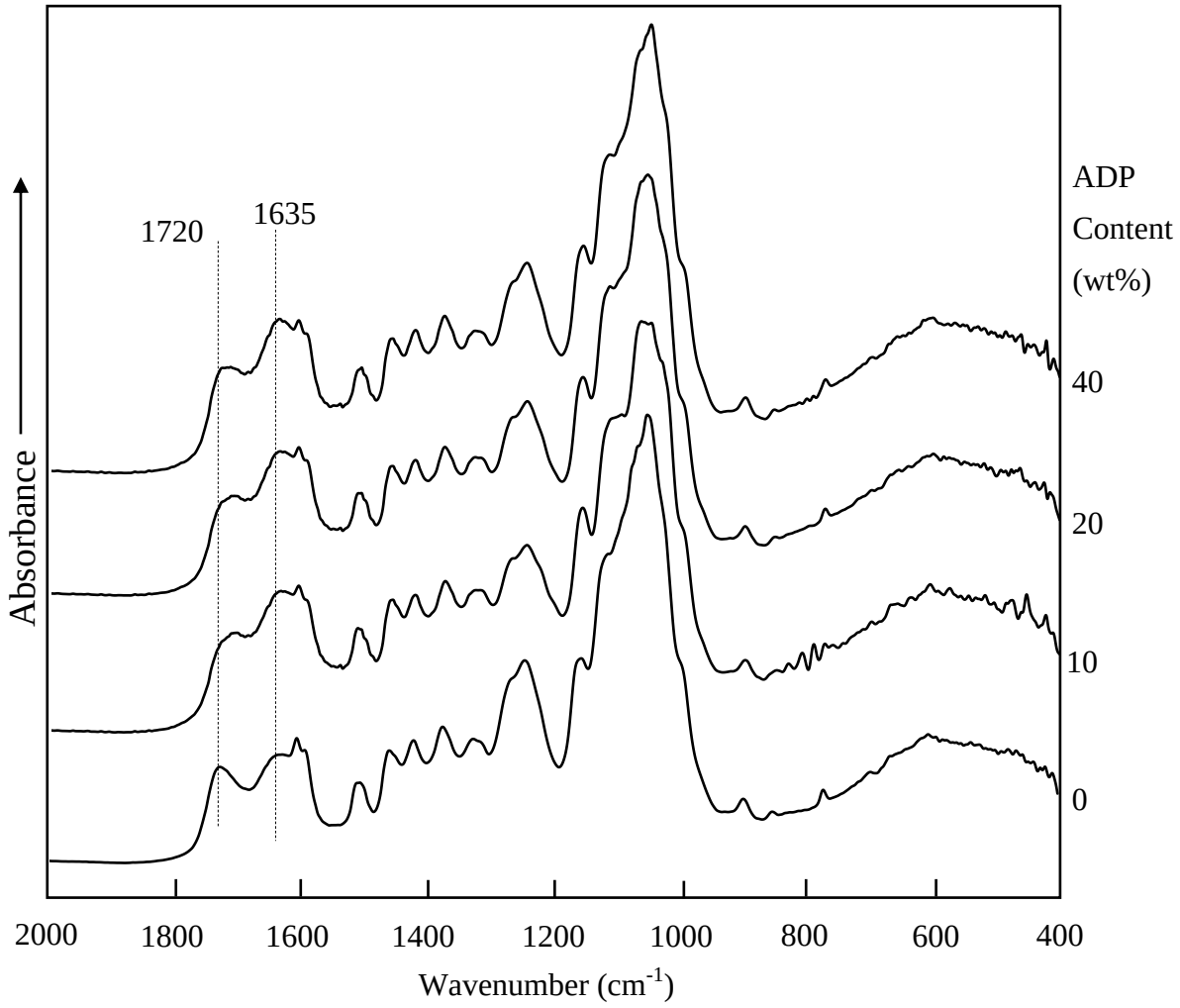



**Fig. 2.11.** FTIR spectra of particleboards on different ADP content (wt%) after cyclic aging treatment.

To observe the chemical structure of the particleboards that excluded water-soluble components such as remaining ADP and some chemical compounds, the FTIR spectra after cyclic aging treatment was measured. The results are presented in Figure 2.11. The peak at ~1720 $cm^{-1}$ was decreased slightly by the addition of ADP, and the peak at ~1635 $cm^{-1}$ was increased slightly with the increase in ADP content. Based on the research by Okuda *et. al.* (2006), the change of the peak at ~1635 $cm^{-1}$ might be attributed to the bonding ability of the particleboards. The ADP thus seemed to have a

role in the changes of chemical compounds of OPT. However, further chemical analyses are required to determine the detailed chemical changes of the particleboards. The peaks derived from ADP were not observed irrespective of the ADP contents, and this indicated that ADP was practically eluted by the treatment and did not react with chemical compounds of OPT.

### 2.3.7. The effects of ADP on the water-soluble components of OPT

The weight change results in Figure 2.9. indicated that the treatment induced the elution of water-soluble components from the particleboard. Hardly any loss of particles from the particleboard was observed, and the particleboards kept their shape. Table 2.3. summarizes the weight changes of the particleboards at the final step of the cyclic aging treatment. It is shown that the particleboards had various initial moisture content between 4.79 to 6.47% before undergoing cyclic aging treatment. The moisture content was used to estimate the initial weight of the particleboards without moisture content (water); as water intake and release will be occurred during the treatment. And in the final step of the cyclic aging treatment weight of the particleboards was measured. The particleboards with 2 to 10 wt% ADP showed weight changes that were similar to that of the 0 wt% ADP (binderless particleboard). Considering the additional weight and the water solubility of ADP, it suspected that some changes occurred, and the changes can be explained in more detail as follows.

The final weight decrease in the particleboard with 0 wt% ADP was −20.64%. This value contains the moisture content of the specimen before the treatment, in addition to the water-soluble components from OPT. Considering the average moisture content (5.59%) of the specimens before the treatment, the actual final average weight decrease

from water-soluble components was calculated as −16.21%. The average weight of the specimens before and after the treatments without the moisture content are estimated as 10.27 and 8.61 g, respectively.

**Table 2.3.** Weight change of the particleboards at the final of cyclic aging treatment.

| Particleboard type | Moisture content of specimen before cyclic aging treatment (%) | Initial weight of specimen without moisture content (g) | Dry weight of specimen at final step (g) | Weight change of specimen at final step (%) |
|---|---|---|---|---|
| 0 wt% (binderless) | 5.59 | 10.28 | 8.61 | -20.64 |
| 2 wt% ADP | 4.79 | 10.16 | 8.54 | -19.80 |
| 5 wt% ADP | 4.86 | 9.44 | 7.89 | -20.30 |
| 10 wt% ADP | 5.97 | 9.96 | 8.04 | -23.86 |
| 20 wt% ADP | 6.47 | 9.79 | 7.29 | -30.09 |
| 30 wt% ADP | 6.12 | 9.61 | 6.76 | -33.69 |
| 40 wt% ADP | 5.61 | 9.68 | 6.28 | -38.55 |

Similarly, as the average moisture content of the specimens of particleboard with 10 wt% ADP before the treatment was 5.97%, the actual final weight decrease was −19.32%. In this case, the decrease is derived from the water-soluble components of OPT and the added ADP. Based on the FTIR results presented in Figure 2.11, ADP was practically eluted by the treatment. The average final weight of the specimens after the treatment was calculated as 8.04 g, which shows only the weight from OPT. On the other hand, the average weights of OPT and ADP in the specimens before treatment are calculated as 9.06 and 0.91 g, respectively. The weight decrease of OPT from 9.06 to 8.04 g is estimated as −11.25%, which is 4.96% different compared to the value (−16.21%) of

particleboard with 0 wt% ADP (binderless particleboard). This means that the elution of OPT by the treatment was inhibited by the addition of ADP. In particularly, this result indicates that the water-soluble components of OPT were changed to water-insoluble components by the addition of ADP. Fig. 2.12 shows the details to understanding the phenomenon that occurs in the particleboard.

Based on the estimation method, the rate of water-insoluble components with the addition of ADP to particleboard with 0 wt% ADP (binderless particleboard) was calculated; the results are shown in Figure 2.13. The rate increased with an increase in ADP contents. Considering the research of Umemura *et. al.* (2017), water-soluble saccharides of the OPT might be changed to water-insoluble components due to the addition of ADP to the manufacturing of the particleboards. The change seemed to contribute to the improvement of the ADP-added particleboards' mechanical properties and water resistance. However, the addition of a high amount of ADP did not contribute to the improvement of these properties because of the low proportion of OPT particles in the particleboard.

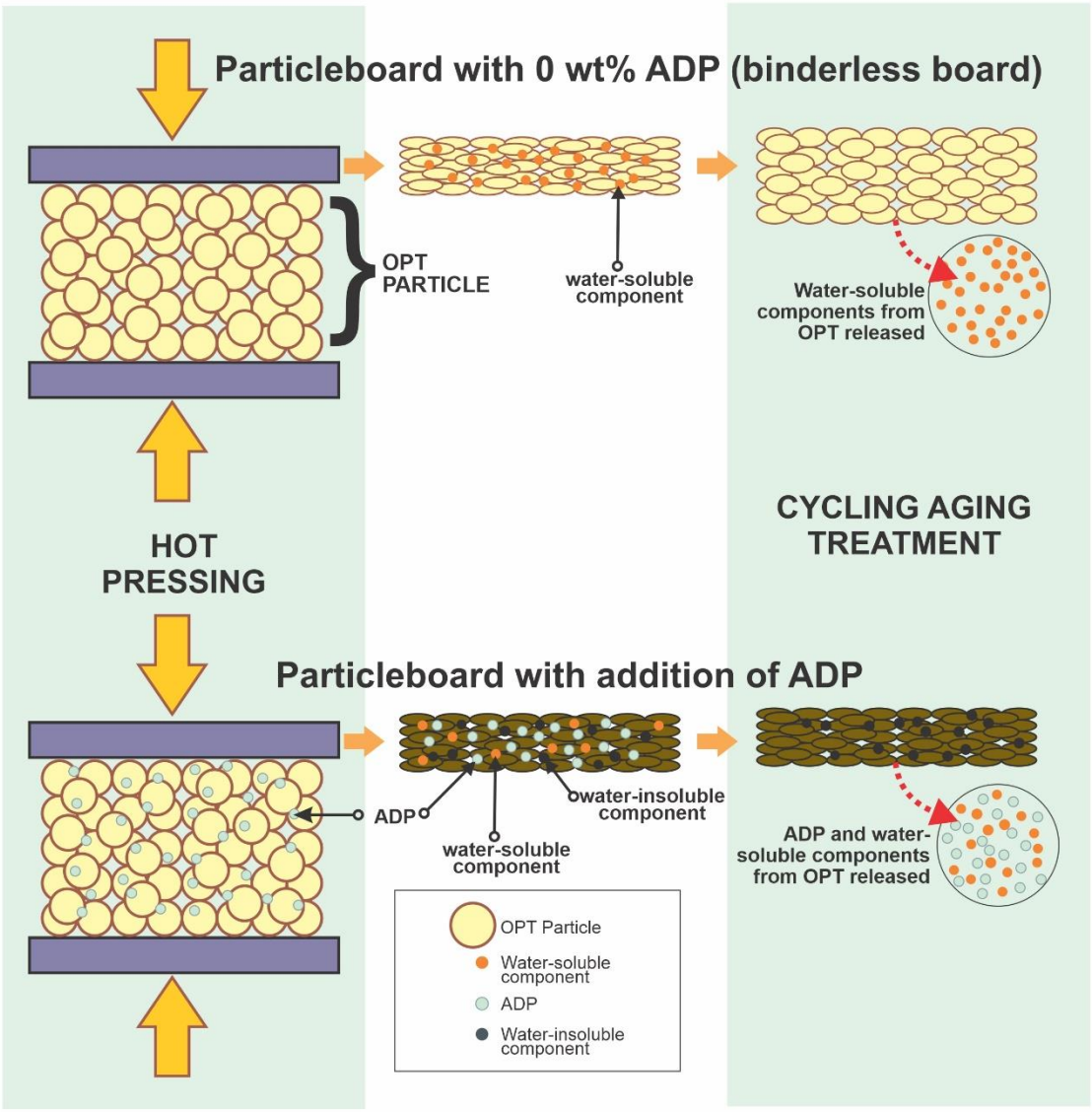



**Fig. 2.12.** Illustration of the phenomenon occurred on the particleboards.

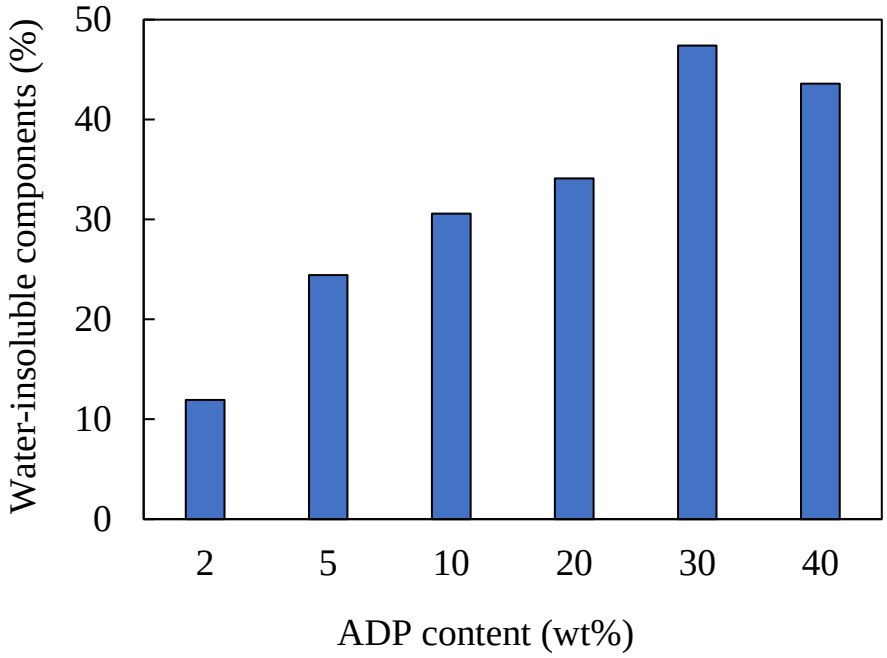



**Fig. 2.13.** The rate of water-insoluble components by the addition of ADP based on particleboard with 0 wt% ADP.

## 2.4. Summary

The effects of adding ADP on the characteristics of particleboard manufactured using the inner part of OPT were investigated. With an increase in the ADP content, the thermal properties of the OPT were significantly lowered. Particleboards were manufactured based on the results of the thermal analyses. The mechanical and physical properties were improved with the addition of ADP until 10 wt%. The particleboard with 10 wt% ADP showed the best values for MOR (8.9 MPa), IB (0.53 MPa), and TS (5.9%). In addition, the particleboards made with ≥10 wt% ADP had excellent water resistance. Based on the FTIR results, a change of the carbonyl group was observed, and the existence of ADP as a water-soluble component was recognized. According to the result of cyclic aging treatment, it was suggested that water-soluble components of OPT

changed to water-insoluble components by the addition of ADP. Consequently, the addition of ADP was effective for improving the physical and mechanical properties of particleboard using the inner part of OPT.

# Chapter 3

## *Influences of ammonium dihydrogen phosphate on the water-soluble extractive of the inner part of oil palm trunk and its binderless particleboard*

### 3.1. Introduction

In Chapter 2, binderless particleboard was successfully manufactured from inner part of OPT. The results showed that ADP was effective for improving the physical and mechanical properties of binderless particleboard using the inner part of OPT, especially excellent water resistance. The free sugar contained in the water-soluble extractive is thought to affect ADP's contribution to the qualities of binderless particleboard. However, the effect of OPT's water-soluble component on the bonding mechanism of binderless particleboard with the added ADP has not been fully clarified.

Therefore, in this chapter, the ADP's influences on the physical and chemical changes of a water-soluble extractive of the inner part of OPT were focused on. The effect of the water-soluble extractive on binderless particleboard with the addition of ADP was also investigated in this chapter using the similar methods that was described in Chapter 2.

## 3.2. Materials and methods

### 3.2.1. Preparation of materials

The raw materials and the preparation were same as mentioned in Chapter 2.

### 3.2.2. Hot-water extraction treatment

To obtain a water-soluble extractive of OPT particles, the particles were immersed in hot water at 60 °C for 6 h (Lamaming *et al*. 2013). The mean concentration was 100 g air-dried weight (~7%) of OPT particles per liter of water. The solution was filtered using a Buchner funnel with medium-fine filter paper. The filtrate was freeze-dried to obtain a water-soluble extractive. The treated particles were oven-dried at 105 °C for 24 h. The particles were used as raw material for manufacturing the binderless particleboard. The details procedure of the experiment is shown in Fig. 3.1.

### 3.2.3. Sugar analyses in extractive

The free sugars of glucose, sucrose, and fructose were quantified in the water-soluble OPT extractive. Briefly, the dried extractive was re-solubilized in distilled water (10 mg/mL) and submitted to the assays using a glucose CII test kit (Wako Pure Chemical, Osaka, Japan), a sucrose assay kit SCA 2-1 KIT, 20 assays (Sigma-Aldrich, St. Louis,

MO), and a fructose assay kit (C/F) (Bio-vision, Palo Alto, CA). Absorbance was measured with an SH-1000 Lab Microplate Reader (Corona Electric, Ibaraki, Japan). Glucose, sucrose, and fructose solutions were used as standards for calibration.

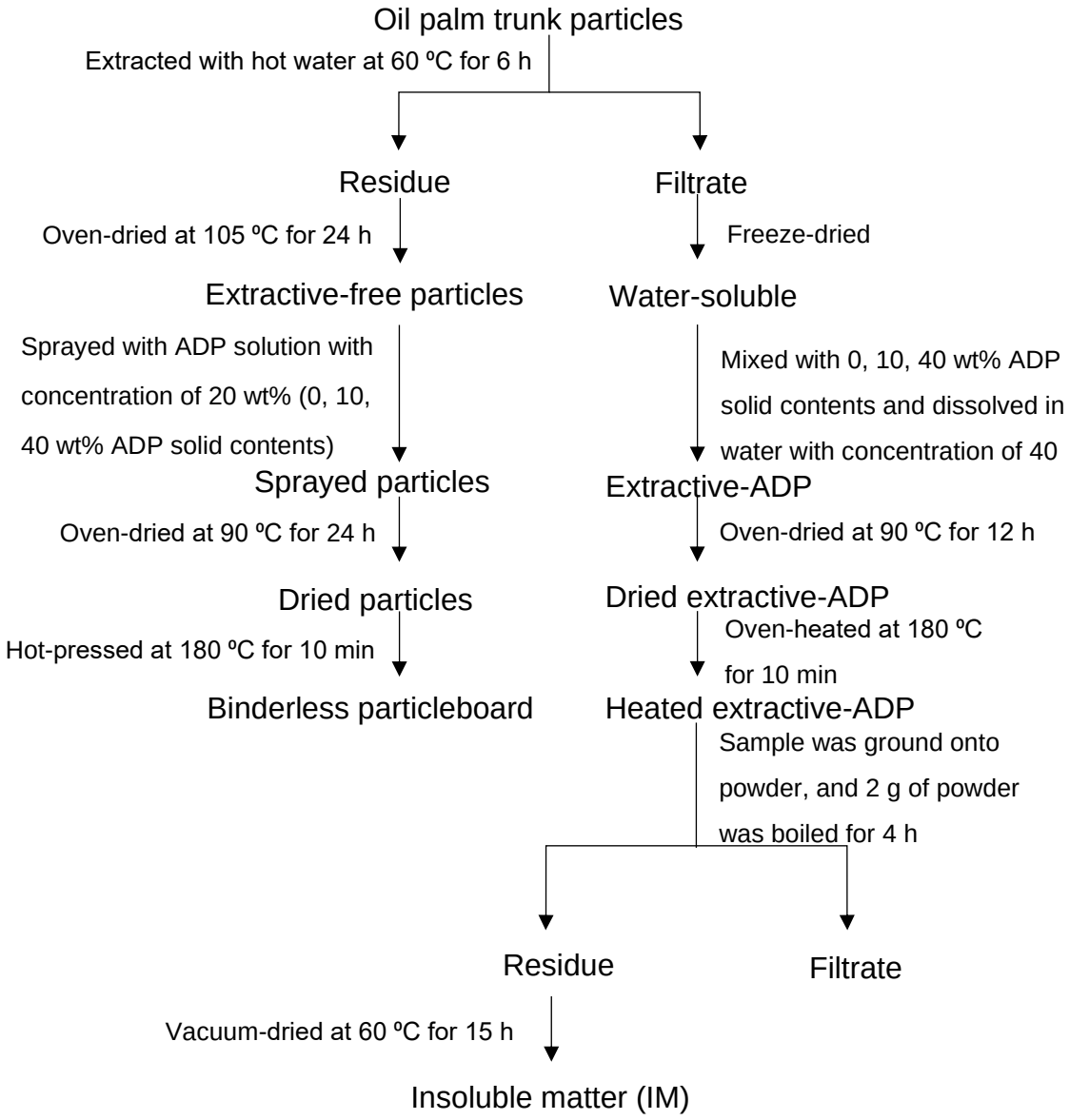



**Fig. 3.1.** Schematic diagram of experiment procedure.

### 3.2.4. Insoluble matter (IM) measurement

The extractive was mixed with various ADP solid contents, i.e., 0, 10, and 40 wt% based on the weight of the dried extractive. After being dissolved in distilled water with a concentration of 40 wt%, the mixtures were oven-dried at 90 ℃ for 12 h then heated at 180 °C for 10 min. The heat-treated mixtures were ground into powder using a mortar. Two grams of powder of each heat-treated mixture was boiled for 4 h. The results were obtained by three repetitions with the same sample. After the treatment, the solution was filtrated with a glass filter, and the insoluble matter remaining on the glass filter was vacuum-dried at 60 °C for 15 h and finally weighed. The IM was calculated based on the weight change of the mixture by the following equation:

$$IM\ rate\ (\%) = \frac{a}{b} \times 100 \quad (3\text{-}1)$$

Where $a$ is weight of dried insoluble matter (g) and $b$ is heat-treated mixture (g).

### 3.2.5. Fourier transform infrared spectroscopy (FTIR) measurement

FTIR analysis was performed using OPT particles, water-soluble extractive, and IM samples. The powders for FTIR analysis were prepared as same as in Chapter 2. The running method of FTIR analysis was explained in Chapter 2.

### 3.2.6. Thermal analysis measurement

OPT particles, OPT extractive, and IM samples were pulverized (passed 150 μm). All of the samples were vacuum-dried at 60 °C for 15 h before analysis. Differential scanning calorimetry (DSC) was carried out with a DSC 25 differential scanning calorimeter (TA Instruments, Tokyo). Before the sample was prepared, tweezers were used to poke a small hole in the cover of an aluminum lid. Each sample for the DSC analysis was encapsulated in an aluminum pan and was scanned from room temperature to 400 °C at an increase rate of 10 °C/min under nitrogen purging with a flow rate of 50 mL/min. Thermogravimetric analysis (TGA) was carried out using a TGA 55 thermogravimetric analyzer (TA Instruments, Tokyo). Each sample of TGA analysis was scanned from room temperature to 400 °C under nitrogen purging with a flow rate of 50 mL/min and an increase rate of 10 °C/min.

### 3.2.7. Binderless particleboard manufacturing

The 20 wt% ADP solution was sprayed onto the hot-water-treated OPT particles with the various ADP solid contents, i.e., 0, 10, and 40 wt% based on the weight of the dried particles. The sprayed particles were dried at 90 °C for 24 h to reduce the moisture content to around 1 to 3%. Subsequently, the particles were formed into mats as same as

in Chapter 2. Furthermore, the detail process of manufacture of particleboards as similar as in Chapter 2.

### 3.2.8. Particleboards properties

The particleboards were tested as same as in Chapter 2. The procedure of preparing sample specimens form each particleboard was as same as in Chapter 2. The physical and mechanical properties were evaluated by thickness swelling (TS), bending, and internal bond strength (IB) tests according to JIS A 5908 (2003). Moreover, water absorbance (WA) was measured and cyclic aging treatment was conducted as explained detail in Chapter 2.

Each test was carried out in five replications, and the average values and standard deviation were calculated. The MOR, MOE, and IB values of the boards were corrected for each target density based on the regression lines between the obtained values and the sample densities.

### 3.2.9. Statistical analysis

The data were statistically analyzed using analysis of variance. The comparison of means was performed using Tukey's HSD post-hoc test to determine the significance

of differences between each group at the 95% confidence level. The standard deviation is also calculated from the data and is represented by error bars in each respective figure.

## 3.3.Results and discussion

### 3.3.1. Free sugars in water-soluble extractive

**Table 3.1.** Free-sugar content in the water-soluble extractive of the inner part of OPT.

| | Glucose (%) | Sucrose (%) | Fructose (%) |
|---|---|---|---|
| Hot-water extraction | 13.63 (0.30) | 2.32 (0.24) | 3.93 (0.80) |

*n=3; value in parentheses are standard deviation

In this study, 11.59% of the water-soluble extractive was obtained. Table 3.1 shows the free sugars in the extractive. The glucose content was the highest, at 13.63%, followed by fructose and sucrose, with 3.93% and 2.32%, respectively. These obtained values were different from those obtained from chemical composition and free sugar analysis in the previous study (Komariah *et al*. 2019). This difference can be explained as a function of raw material size and to the difference in the methods used to obtain the extractive. In this study, the particles were extracted directly with hot water, whereas in the previous study the raw material was a powder. The solvents used to obtain free sugars also differed between studies. However, the present study's results are consistent with

those of Lange and Simatupang (1994) and Murai and Kondo (2011), in which glucose had the highest sugar component in the hot-water extractive of OPT particles.

### 3.3.2. IM of extractive with ADP added

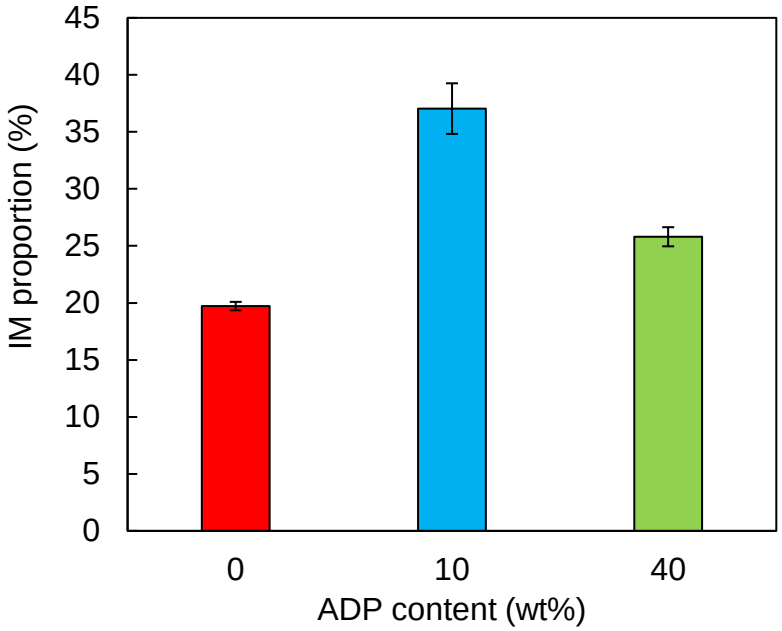



**Fig. 3.2.** Insoluble matter (IM) of OPT with added ADP.

To clarify the chemical and physical changes resulting from the addition of ADP to the water-soluble extractive, a mixture of extractive and ADP was heated and treated by boiling water. Figure 3.2 shows the IM proportion against boiling water. The IM proportion without ADP (0 wt%) was 19.72%. This means that the extractive turned into IM by only heating. When 10 wt% of ADP was added to the extractive, the IM proportion almost doubled, to 37.04%. In the case of 40 wt% ADP, the proportion was 25.80%. The

reason why the value was lower than that of 10 wt% seems to be that a large amount of ADP was eluted during treatment. Meanwhile, when the practical IM was calculated based on the weight of the extractive, the IM proportion of 40 wt% ADP was higher than that of 10 wt% ADP: 43.58% vs. 38.31%, respectively. It was clarified that ADP accelerates the change of water-soluble extractive into an insoluble substance. This is in accordance with Umemura *et al*. (2017) and Zhao *et al*. (2018), who stated ADP content of 10 wt% was needed to obtain a highly insoluble substance from sucrose.

### 3.3.3. FTIR measurement

The FTIR spectra of OPT particles, OPT extractive, and each IM are shown in Fig. 3.3. In the OPT particles, seven characteristic absorption peaks were observed at 1630, 1515, 1405, 1361, 1241, 1077, and 1051 $cm^{-1}$. The absorption at 1630 $cm^{-1}$ is probably associated with the C=O stretch of hemicelluloses (Sun *et al*. 1999). The peak at around 1515 $cm^{-1}$ indicates C=C stretching of the aromatic ring (lignin) (Lee *et al*. 2018). The peaks at 1405, 1361, and 1241 $cm^{-1}$ represent O-H and C-H bendings (Sun *et al*. 1999). The peaks at 1077 and 1051 $cm^{-1}$ are attributed to the C-OH bending from hydroxyl groups in polysaccharides (Kacurakova *et al*. 1994). In the OPT extractive, all the peaks except for 1515 $cm^{-1}$ were observed. This means that OPT extractive showed

similar hemicellulose-indicative absorbances to OPT particles but did not show lignin-related absorbance.

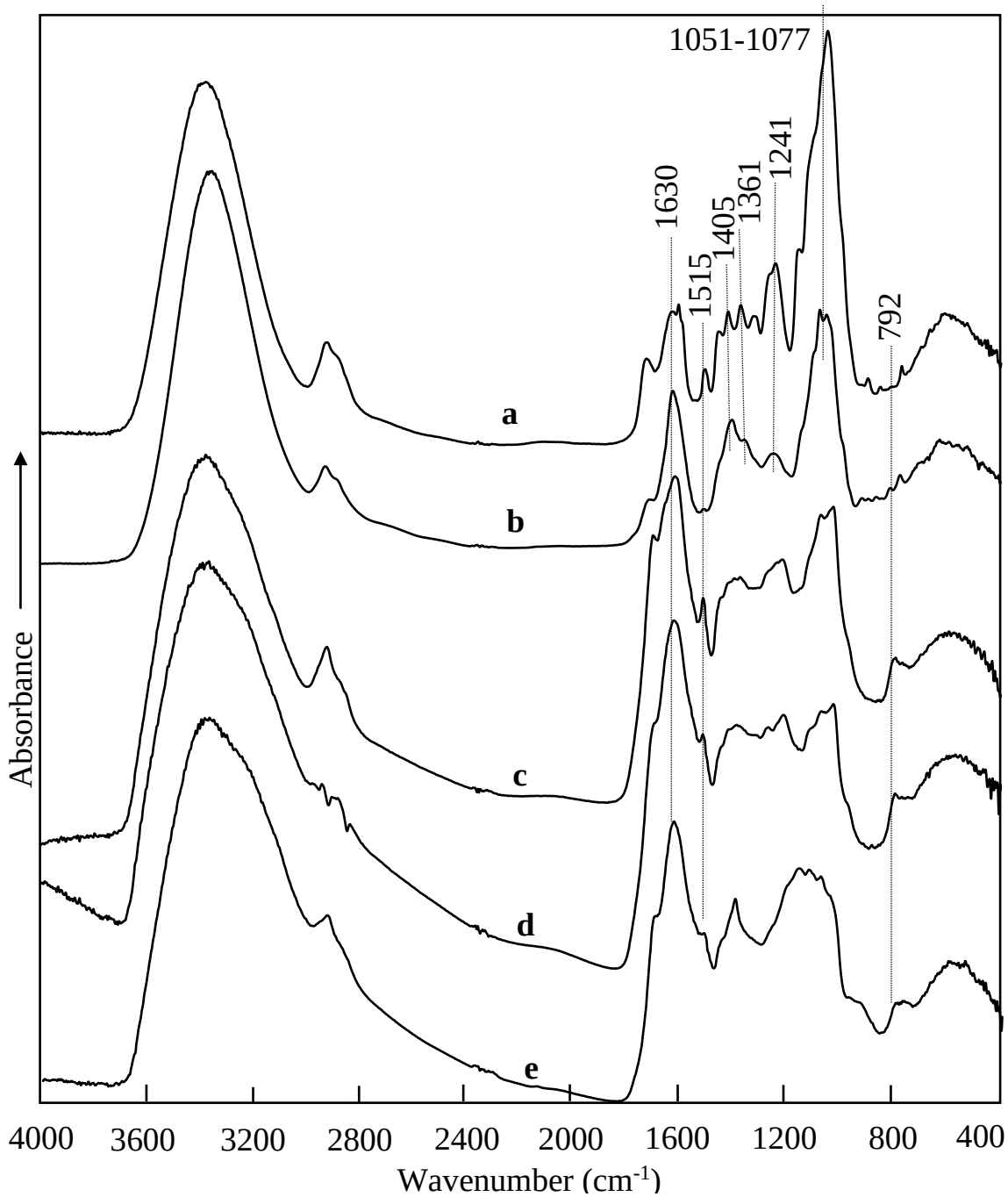



**Fig. 3.3.** FTIR spectra of (a) OPT particles, (b) OPT extractive, (c) IM of OPT with 0 wt% ADP, (d) IM of OPT with 10 wt% ADP, and (e) IM of OPT with 40 wt% ADP.

However, the addition of ADP to OPT extractive after boiling treatment (IM) showed two new peaks, at around 1515 and 792 $cm^{-1}$, that were not found in the OPT extractive spectra. Umemura *et al.* (2017) attributed the peak around 1510 $cm^{-1}$ to C=C of the furan ring. Also, Sun *et al*. (2019) attributed the peak at around 780 $cm^{-1}$ to CH=CH of the furan ring, and this observation might be related to the production of 5-hydroxymethylfurfural (5-HMF) from the sugar compound on the OPT extractive to form a furan polymer. Therefore, the peaks at around 1515 and 792 $cm^{-1}$ might be attributable to the formation of the furan ring in the IM. Also, the peak at around 1630 $cm^{-1}$ showed increasing absorbance intensity in all IM samples irrespective of ADP content.

The FTIR results showed that the IM contained a furan ring and a carbonyl group. Therefore, it was considered that the water-soluble component was easy to change into insoluble matter containing furan and carbonyl compounds due to heating and to the addition of ADP.

### 3.3.4. Thermal analysis measurement

The thermal behaviors of OPT particles, OPT extractive, and each IM were analyzed by DSC and TGA. The results of DSC are shown in Fig. 3.4. The DSC curve for the OPT particle had one endothermic peak at around 70 °C and two broad exothermic

peaks at approx. 270 and 330 °C. Meanwhile, the DSC curve for the OPT extractive had two endothermic peaks at around 85 and 135 °C and one broad exothermic peak at around 230 °C. The DSC curves of the mixtures of the various IMs were similar regardless of the ADP content; each curve showed two endothermic peaks at around 110 and 230 °C.

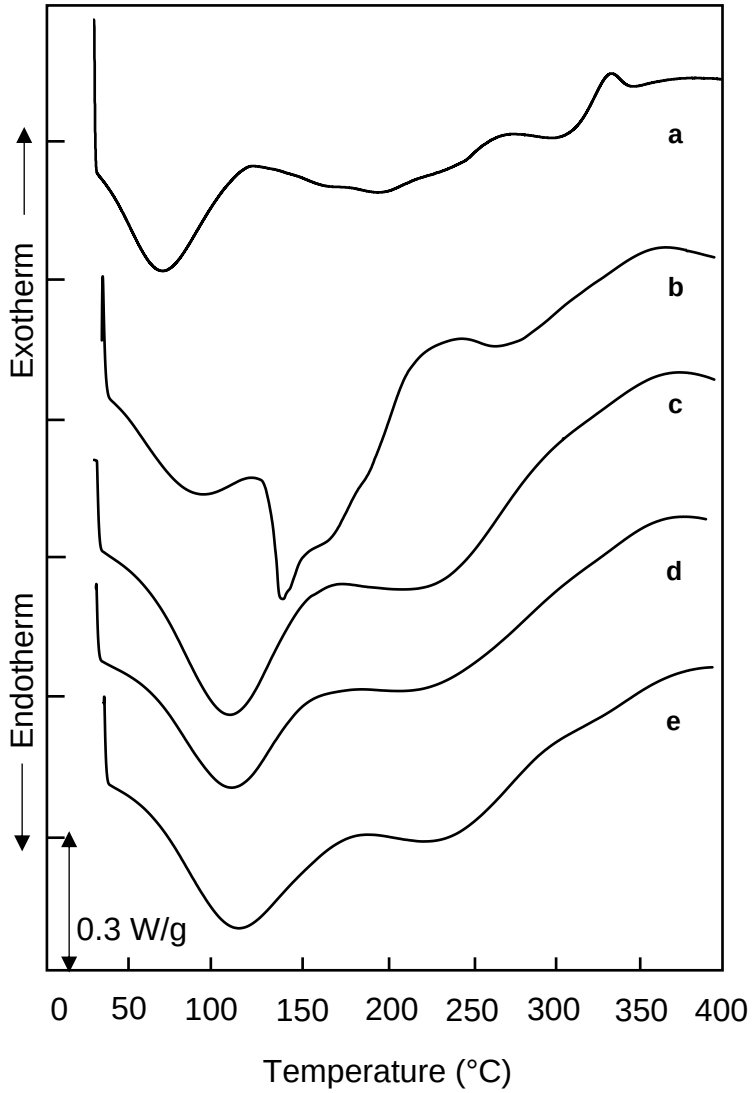



**Fig. 3.4.** DSC graph of (a) OPT particles, (b) OPT extractive, (c) IM of OPT with 0 wt% ADP, (d) IM of OPT with 10 wt% ADP, and (e) IM of OPT with 40 wt% ADP.

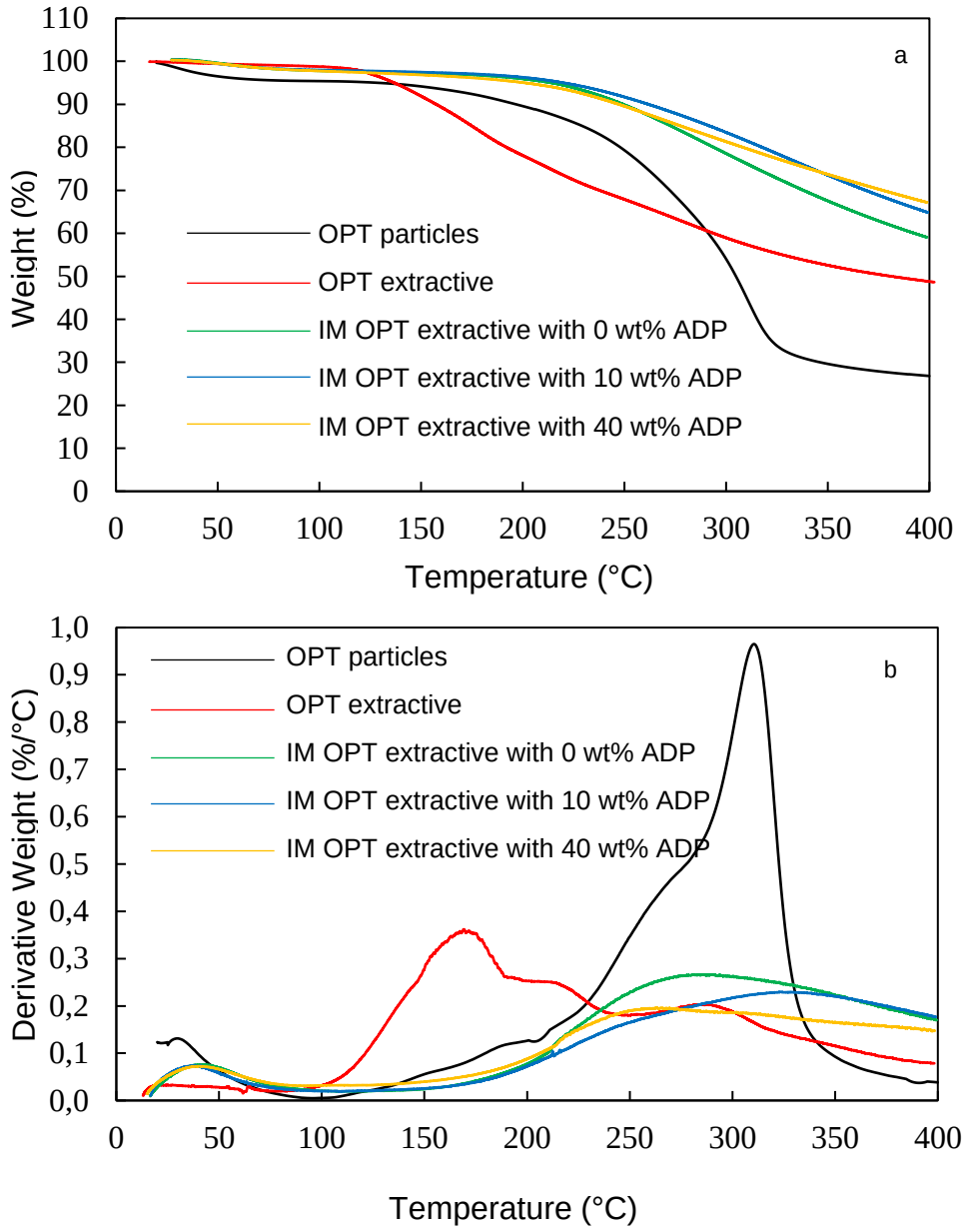



**Fig. 3.5.** Thermogravimetric (TG) (a) and Derivative TG (DTG) (b) graphs of OPT particles, OPT extractive, and IM of OPT with added ADP (wt%).

The results of thermogravimetric (TG) and derivative TG (DTG) analyses are shown in Fig. 3.5. In the TG curves (a), the OPT particles showed reduced weight at temperatures between 200 and 330 °C. According to the DTG curves (b), the OPT

particles lost significant weight at ~310 °C. The onset temperature of weight reduction of the OPT extractive was about 135 °C (a), and three-step weight reduction was observed in the DTG curve (b): the first step at around 170 °C, the second at around 215 °C, and the third at around 275 °C. Meanwhile, the IM results had similar weight-reduction onset temperatures at around 200 °C, and one broad weight reduction that started around 200 °C (a) was observed in the DTG curve (b) irrespective of the ADP content.

The thermal behaviors of OPT particles and OPT extractive differed due to the differences in chemical compounds, as the OPT extractive consisted of low-molecular-weight compounds compared to OPT particles. The endothermic peaks of DSC of around 70 ~ 85 °C observed in OPT particles and OPT extractive indicated the weakening effect of hydrogen bonds between carbohydrates (Mehrotra *et al*. 2010); this was supported by the fact that the TG and DTG curves that did not show clear weight reduction in that temperature range. The exothermic peak at around 270°C on OPT particles would be attributable to the decomposition of hemicellulose and the slower decomposition of lignin, according to Yang *et al*. (2007). Shebani *et al*. (2008) suggested that the exothermic peak at around 330 °C indicates the decomposition of cellulose. These phenomena were also supported by the weight reduction between 200 and 330 °C in TG of OPT particles. According to Hurtta *et al.* (2004), the melting peak temperatures of fructose and glucose

were around 131 and 159 °C, respectively. This was similar to the finding of an endothermic peak at around 135 °C on the OPT extractive; wherein it shows in Table 1 that the OPT extractive contains glucose and fructose.

The DSC and TGA of the IM mixtures showed similar behaviors regardless of ADP content. They showed the upward shifts in the onset temperature and weight reduction temperature of the IM mixtures compared to the OPT extractive and the OPT particles. These results indicated that the OPT extractive incurred changes in some high-molecular-weight substances due to heating and ADP addition, as indicated by the higher temperature of weight reduction. As a result, the IM mixtures had good thermal stability that contained high-molecular-weight substances.

### 3.3.5. Water resistance properties of binderless particleboard using hot-water extracted particles

To investigate the effects of the water-soluble extractive on binderless particleboards with ADP added, boards were manufactured using hot-water-treated particles. Figure 3.6 shows the TS and WA of the particleboards at various ADP contents. The water resistance of the particleboards improved with the addition of ADP. The TS values of the particleboard without ADP addition (0 wt%) was 140%. The addition of

ADP significantly decreased the TS values, which were 33% and 28% for 10 wt% and 40 wt% ADP additions, respectively. This was different from our previous study (Komariah *et al*. 2019), in which binderless particleboard (without ADP addition) using untreated particles had a TS value of 49%. The difference was similar to that reported by Lamaming *et al*. (2014), in which an OPT binderless board with hot-water-treated particles had a higher TS value than boards made with untreated particles.

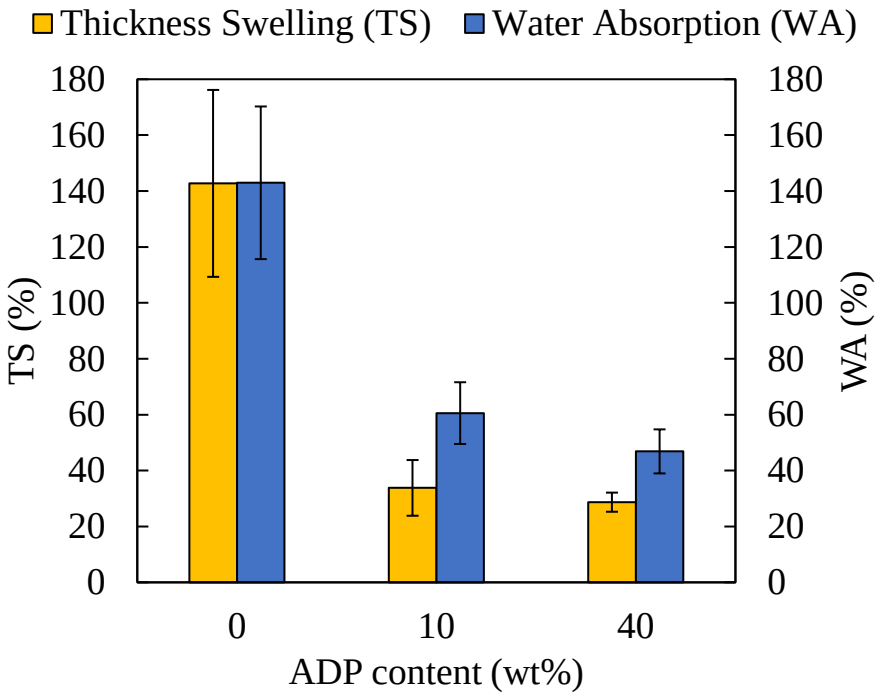



**Fig. 3.6.** Thickness swelling and water absorption of the binderless particleboards.

The same trend observed for TS was also seen for WA values. The WA value of the particleboard without ADP addition (0 wt%) was 142%, whereas the addition of ADP 10 wt% and 40 wt% resulted in significantly decreased values, to 60.55% and 46.86%,

respectively. Meanwhile Komariah *et al*. (2019) showed that binderless particleboard (without ADP addition) using untreated particles had a WA value of about 65%. Interestingly, the 10 and 40 wt% ADP did not differ significantly ($p>0.05$) in TS or WA values. This indicated that the addition of ADP played an important role in increasing water resistance regardless of the amount added. However, compared to previous study (Komariah *et al*. 2019), the TS and WA values of treated particles were higher than those of untreated particles.

The changes in thickness and weight by the cyclic aging treatment were observed further to investigate the particleboard's water resistance under severe conditions. The results are shown in Figure 3.7. The extent of the thickness change at each stage of the cyclic aging treatment decreased with the addition of ADP. The specimens with ADP maintained their shapes throughout the stages of the cylic aging treatment. However, the specimen without ADP (0 wt%) decomposed while being boiled in water for 4 h. The thickness change values of particleboards with 10 and 40 wt% ADP addition in the final stage of cyclic aging treatment were 32.7 and 43.0%, respectively. These values were higher than those of Komariah *et al*. (2019), which were about 0.2 and 8.63%, respectively, when using untreated particles. The weight changes of the particleboards

also decreased with the addition of ADP, regardless of ADP content, compared to that without ADP addition.

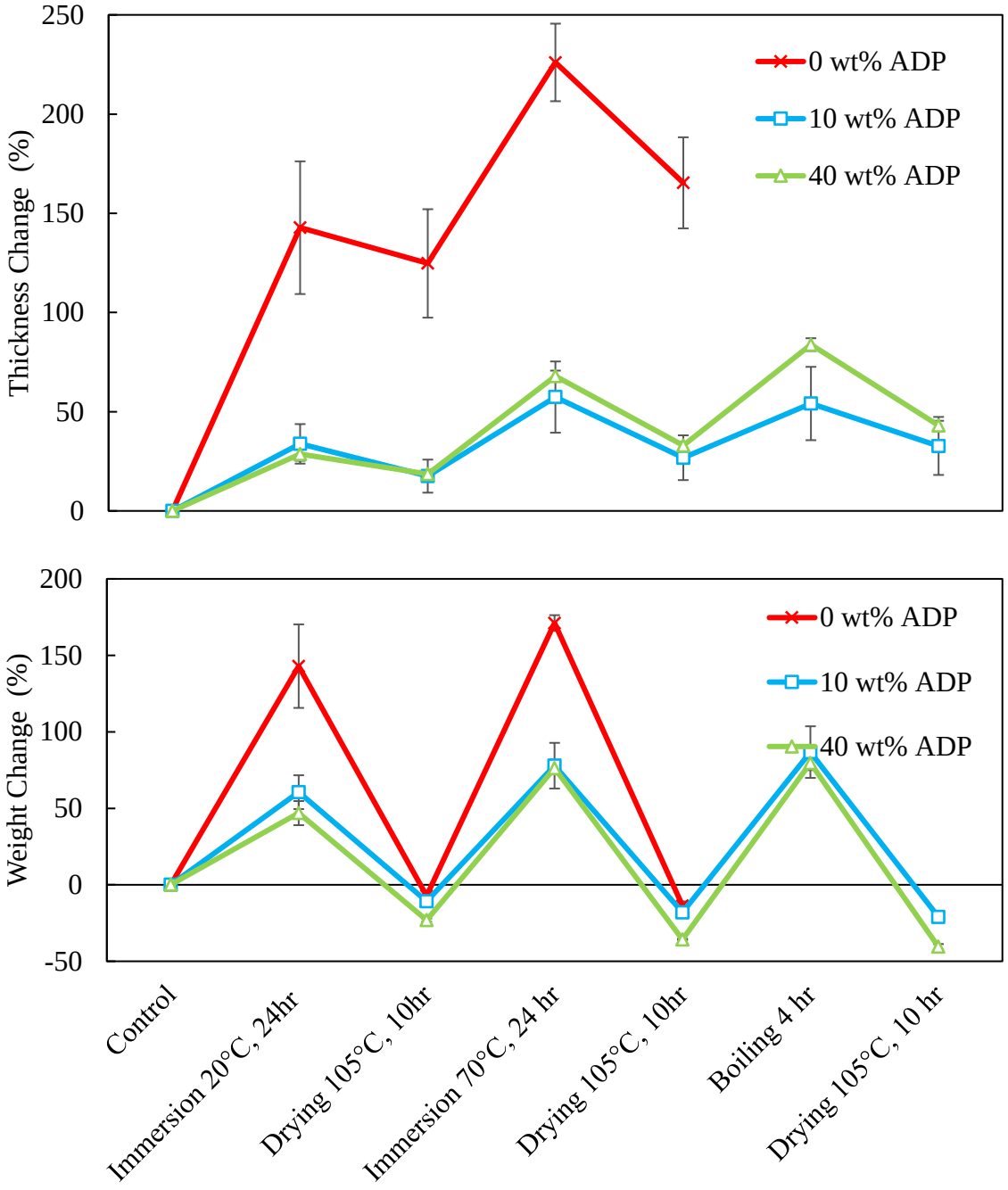



**Fig. 3.7.** Thickness and weight changes of the binderless particleboards in the cyclic aging treatment.

The results showed the importance of extractive consisting of components that prevent water absorption. In this case, when the extractive was removed, ADP would decompose the chemical components of the OPT particles to some degree, which subsequently turned into an insoluble component that prevented water absorption, as shown in the TS value (Fig. 3.6). In other words, the addition of ADP and the existence of the extractive are important in the manufacturing of water-resistant particleboard.

### 3.3.6. Mechanical properties of binderless particleboards

Figure 3.8 shows the bending properties of the particleboards with ADP addition. The MOR value of the particleboard without ADP (0 wt%) was 2.80 MPa. The MOR values approximately tripled when 10 wt% ADP content was added compared to the particleboard without ADP. The addition of 40 wt% ADP showed a slightly lower MOR value. This was due to the excess of ADP that caused a decrease in particle proportion and thus did not contribute to the bondability of the particleboards. The performance of the particleboard without ADP was similar to the result of Lamaming *et al*. (2014) using treated particles; the MOR value was 3.92 MPa. However, this value was lower than those in Komariah *et al*. (2019) and Lamaming *et al*. (2014), which were 5.23 and 5.25 MPa, respectively, with untreated particles. Furthermore, the hot-water treatment of the

particles did not affect the MOR values of the particleboards with ADP addition, as the present results were similar to those in Komariah *et al*. (2019) with untreated particles. The trend with MOR values was also seen in the MOE values of the particleboards, as shown in Fig. 3.8. The MOE value of the particleboard without ADP (0 wt%) was 1.15 GPa. After the addition of ADP, the MOE values doubled. This shows that adding ADP enhanced the strength characteristics of the particleboards using treated OPT particles.

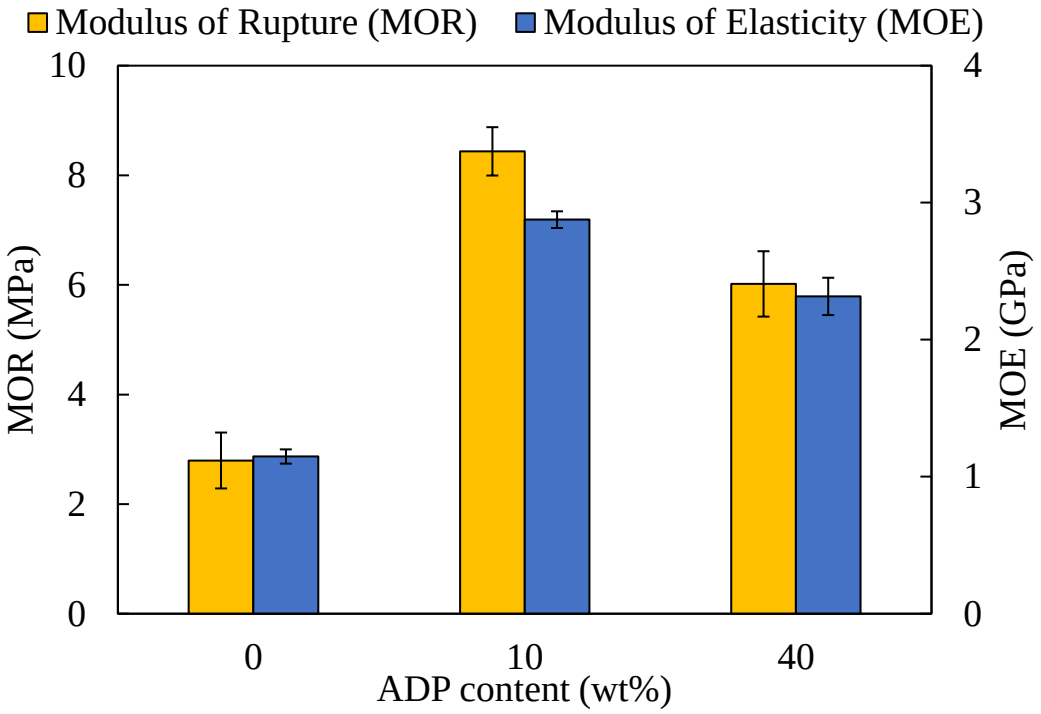



**Fig. 3.8.** Bending properties of the binderless particleboards.

Figure 3.9 shows the IB strength of the binderless particleboards. It can be seen that the IB strength was increased with the addition of 10 wt% ADP. The IB of the

particleboard without ADP (0 wt%) was 0.38 MPa, similar to the result of Komariah *et al*. (2019) with untreated particles. This result showed that the hot-water treatment hardly affected the IB strength of binderless particleboard. In addition, the IB strength of binderless particleboard with the addition of 10 wt% ADP was increased by 70%, to 0.53 MPa. Judging from the results obtained, the addition of 10 wt% ADP could improve the mechanical properties of particleboards using treated particles.

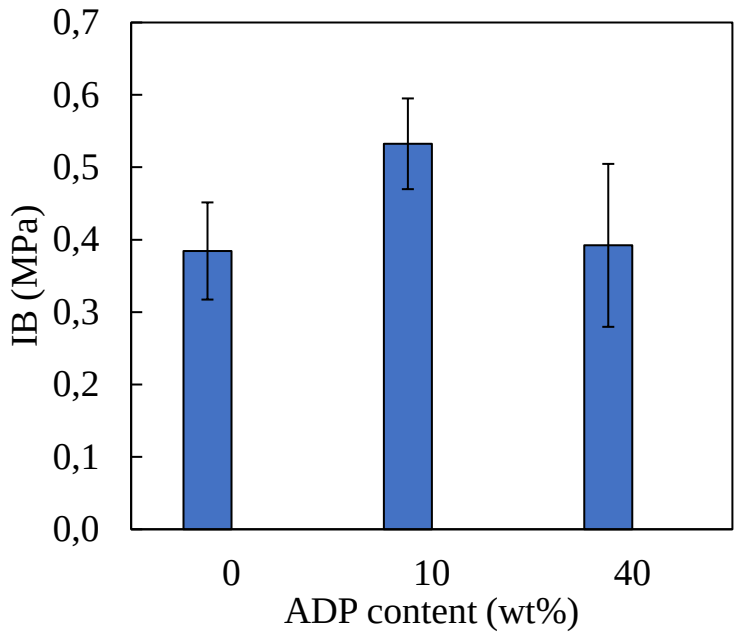



**Fig. 3.9.** Internal bond (IB) of the binderless particleboards.

### 3.4. Summary

The influences of adding ADP on the physical and chemical changes of a water-soluble extractive of the inner part of OPT were clarified. With heating, the extractive changed into an insoluble substance, and the addition of 10 wt% ADP doubled the insoluble substance amount. Based on the FTIR results, the formation of furan compounds derived from sugars was inferred. And the thermal analysis showed that the formed insoluble substance had good thermal stability, suggesting a high-molecular-weight substance. Particleboards using treated particles were manufactured. The boards' physical and mechanical properties irrespective of ADP addition tended to be inferior to those of previously reported particleboards without extracted treatment. In particular, the water-soluble extractive was essential for the water resistance of the particleboard. The particleboard with 10 wt% ADP showed the best values for MOR (8.4 MPa), IB (0.53 MPa), and TS (33.8%). In addition, that particleboard had good water resistance in cyclic aging treatment. Therefore, ADP addition was effective for improving particleboard properties even in the absence of water-soluble extractive.

# Chapter 4

## *Characterization of Particleboard Using the Inner Part of Oil Palm Trunk with a Bio-based Adhesive of Sucrose and Ammonium Dihydrogen Phosphate*

### 4.1. Introduction

In Chapter 3, the influences of adding ADP on the physical and chemical changes of a water-soluble extractive of the inner part of OPT were clarified and the contribution of ADP addition on the properties of the inner part of OPT binderless particleboard was confirmed. ADP changed water-soluble saccharides to water-insoluble components during hot-pressing (Komariah *et al.* 2019; Komariah *et al.* 2021). This means that saccharides have an important role in the properties of particleboards made from the inner part of OPT.

Various recent studies have indicated that sucrose-based adhesives have advantages for composite products. Sucrose is a common disaccharide sweetener in the food and beverage industries, and constitutes an abundantly available, and a bio-resource. Lamaming *et al.* (2014) showed that the addition of sucrose could improve the physical and mechanical properties of binderless particleboard made from oil palm trunks. Sucrose reacts well with citric acid (Umemura *et al.* 2013; Kusumah *et al.* 2017; Sun *et al.* 2019;

Zhao *et al.* 2019b) and tannin (Zhao & Umemura 2014; Sun & Zhao 2018) to produce good bondability in wood composite products. After heating, structures in the sucrose convert to furan compounds that are predicted to contribute to its bonding action as an adhesive for composites. Previously, Umemura *et al.* (2017) found that with the addition of ADP and treatment with heat, sucrose could be transformed into a high-water-resistance substance and suggested that sucrose with added ADP would likely be a water-resistant wood adhesive. Zhao *et al.* (2018) applied a mixture of sucrose and ADP to a wood particleboard manufacturing. The results showed that the particleboard achieved good mechanical and physical properties, especially excellent water resistance. Furthermore, Zhao *et al.* (2019a) proposed that the main reactions during the curing process were the dehydration condensation of furan compounds and the Maillard reaction. In addition, Widyorini (2020) showed that in petung bamboo particleboard manufacturing, the optimal ratio of sucrose to ADP is 95/5 (wt%/wt%).

Considering these previous findings, it could be hypothesized that adding sucrose with ADP as an adhesive might improve the properties of OPT particleboard. It is expected that the addition of sucrose to have a synergistic effect with the saccharide components of the OPT. Therefore, this chapter investigated the effects of various ratios of sucrose and ADP and different resin contents on particleboard's mechanical and

physical properties made of OPT's inner part. In addition, the termite and decay resistance and the particleboard's infrared spectra (IR) were also examined.

## 4.2. Materials and methods

### 4.2.1. Preparation of materials

The inner part of OPT particles were prepared as same as in Chapters 2 and 3. Sucrose and ammonium dihydrogen phosphate (ADP) of extra-pure grade were purchased from Nacalai Tesque (Kyoto, Japan) and used without further purification. Different weight-ratio mixtures of sucrose and ADP (Table 4.1) were dissolved in distilled water at the concentration of 50 wt%, and those mixture solutions were used as adhesives. The pH and viscosity of the adhesives were measured using a pH meter (D - 51 Horiba, Tokyo) and viscosity meter (Fungilab, Barcelona, Spain), respectively. PF resin (B-1370 type) and pMDI (B-1605 type) from Oshika Co. Ltd. (Tokyo, Japan) were used as reference adhesives.

**Table 4.1.** Viscosity and pH of sucrose-ADP adhesives.

| Mixture ratio of sucrose to ADP (wt%) | Concentration (wt%) | Viscosity (mPa•s) | pH |
|---|---|---|---|
| 95:5 | 50 | 22.0 | 3.93 |
| 90:10 | | 21.0 | 3.95 |
| 85:15 | | 20.5 | 3.89 |
| 80:20 | | 21.0 | 3.90 |
| 75:25 | | 21.5 | 3.89 |
| 70:30 | | 21.0 | 3.73 |
| 60:40 | | 21.0 | 3.73 |
| 50:50 | | 21.0 | 3.70 |

### 4.2.2. Manufacturing of particleboards

Based on the results of the previous study (Zhao *et.al.* 2018), 20 wt% solid content of sucrose-ADP adhesive solution based on the weight of the dried OPT particles was sprayed onto OPT particles. The sprayed particles were then dried at 90 °C for 24 h until the moisture content was <3%. Subsequently, the dried sprayed particles were formed into a mat as same as in Chapters 2 and 3. The particleboards were manufactured under the following two conditions: (a) Mats mixed with adhesive solutions with various sucrose-ADP ratios were hot-pressed at 180 °C for 10 min; (b) Mats were mixed with sucrose-ADP 80:20 adhesive at resin contents of 5-40 wt% (solid basis) were hot-pressed

at 180 °C for 10 min. Details of the manufacturing conditions of the two types of particleboards are shown in Table 4.2.

**Table 4.2.** Particleboards Manufacture Conditions

| Group | Mixture ratio of sucrose to ADP (wt%) | Resin content (wt%) | Hot pressing temperature (°C) | Hot pressing time (min) |
|---|---|---|---|---|
| a | 95:5 | 20 | 180 | 10 |
| | 90:10 | | | |
| | 85:15 | | | |
| | 80:20 | | | |
| | 75:25 | | | |
| | 70:30 | | | |
| | 60:40 | | | |
| | 50:50 | | | |
| b | 80:20 | 5 | 180 | 10 |
| | | 10 | | |
| | | 15 | | |
| | | 20 | | |
| | | 30 | | |
| | | 40 | | |

The detail process of manufacturing of particleboards, include particleboards size and target density that used in this chapter were same as mentioned in Chapters 2 and 3. In addition, particleboards bonded using 15 wt% content of PF resin and 8 wt% content

of pMDI with a size and target density similar to those of particleboards bonded using sucrose-ADP adhesive were manufactured as references. The pressing temperature and time were respectively 140 °C for 6 min for particleboard bonded using PF resin and 180 °C for 4 min for particleboard bonded with pMDI.

### 4.2.3. Evaluation of the particleboards

The particleboards were tested according to JIS A 5908 (2003), same as mentioned in Chapters 2 and 3. A static three-point bending test was carried out on 200 × 30 × 5 mm samples of each type of particleboard. The effective span was 150 mm, and the cross-head speed was 10 mm/min. The modulus of rupture (MOR) and the modulus of elasticity (MOE) were calculated. The internal bond (IB) test was performed on a 50 × 50 × 5 mm specimen with a tension loading speed of 2 mm/min. The thickness swelling (TS) after water immersion at 20 °C for 24 h was measured on specimens with dimensions of 50 × 50 × 5 mm. The water absorption (WA) of the specimens and the pH values of the soaked water were measured. After the TS test, the specimens were subjected to a cyclic aging treatment: drying at 105 °C for 10 h, hot water immersion at 70 °C for 24 h, drying at 105 °C for 10 h, immersion in boiling water for 4 h, and drying at 105 °C for 10 h. Changes in thickness and weight of the specimens throughout the

treatments were observed and recorded. Each test was carried out in five replications, and the average values and standard deviations were calculated. The MOR, MOE, and IB values of the boards were corrected for the target density based on the regression lines between the obtained values and the specimen densities.

### 4.2.4. Statistical analysis

The detail explanation of the statistical analysis was described in Chapters 2 and 3.

### 4.2.5. Termite and decay resistance tests

The termite and decay resistance of selected particleboards were evaluated. The size of specimens used for these tests was 20 × 20 × 5 mm. The termite test for the particleboard was performed according to the Japan Industrial standard JIS K 1571:2010 (2010). The specimens were exposed to the subterranean termite *Coptotermes formosanus* Shiraki that obtained from a laboratory termite colony at Research Institute for Sustainable Humanosphere (RISH), Kyoto University, Japan. Open acrylic cylinders of 80 mm diameter, 60 mm height and a 5-mm-thick hard dental plaster (New Plastone, GC Corp) bottom were used as containers. A single test specimen was set at the center of

the plaster bottom of the test container. A total of 150 worker and 15 soldier termites were introduced into each test container. Small wood blocks (20 × 20 × 5 mm) of sugi (*Cryptomeria japonica*) were used as the control. Five specimens for each board type were assayed against termites. The assembled containers were set up on damp cotton pads to supply water to the specimens and left at 28 °C and >85% RH in darkness for three weeks. The mass loss of the specimens was calculated based on the difference between the initial and final oven-dried (60 °C for 48 h) weights of the specimens.

The decay resistance test was performed according to the JIS K 1571:2010 standard method (2010). The specimens were exposed to two wood-degrading fungi, the brown-rot fungi [*Fomitopsis palustris* (Berk. Et Curt) Gilbn. & Ryv. (FFPRI 0507)] and the white-rot fungi [*Trametes versicolor* (L.:Fr.) Pilat. (FFPRI 1030)]. *F. palustris and T. versicolor* cultures were received from the RISH, Kyoto University, Japan. After measuring their oven-dried (60 °C for 48 h) weights, specimens were sterilized with gaseous ethylene oxide before exposure. The glass jars were prepared to put medium and grow the mycelium. Three specimens were placed on top of the growing mycelium when it had entirely covered the medium. Plastic mesh spacers were placed under the specimens. Small wood blocks (20 × 20 × 5 mm) of sugi (*Cryptomeria japonica*) were used as a control. The test jars were incubated at 27 °C and 70% RH for 12 weeks. Nine replicates

were tested for each decay fungi for each board type. The average mass loss calculated from oven-dry weights of nine specimens before and after the decay procedure.

### 4.2.6. Fourier transform infrared spectroscopy (FTIR) analysis

Fourier transform infrared spectroscopy (FTIR) analysis was performed using specimens after cyclic aging treatment. The procedure and details were similar as in Chapters 2 and 3.

## 4.3. Results and Discussion

### 4.3.1. Effects of mixture ratios of sucrose and ADP

Sucrose-ADP adhesive at various mixture ratios was utilized to manufacture OPT particleboard, and its bonding properties were assessed. First, particleboards were manufactured using similar hot-pressing conditions, 180 °C for 10 min, and a set resin content of 20 wt%. Fig. 4.1 shows the bending properties of the particleboards on various ratio of sucrose-ADP. The bending properties decreased gradually with increasing ADP content. The particleboards with sucrose-ADP ratio of 95:5 had the highest MOR value, 19.02 MPa. However, statistical analysis showed no significant difference ($p>0.05$) from the particleboard with sucrose-ADP ratios of 90:10, 85:15, or 80:20. The MOE value of

particleboards with a sucrose-ADP ratio of 90:10 was the highest although, again, the statistical analysis showed no significant difference ($p>0.05$) from those with 85:15 or 80:20 ratios. The bending properties of particleboard with sucrose-ADP adhesive were significantly improved compared to those of binderless board and ADP particleboard (0:100) manufactured in the previous study (Komariah *et al.* 2019). The sucrose-ADP adhesive nearly tripled the MOR and MOE values of the OPT-based particleboard though these were still lower than the MOR and MOE values of PF and pMDI particleboards, with one exception: the MOE value of PF was lower than that of the sucrose-ADP-based adhesives.

The IB values of the particleboards are shown in Fig. 4.2. The IB strengths of particleboards continuously increased with a decreasing sucrose-ADP ratio to 80:20, where it reached a maximum; from there it decreased with decreasing sucrose-ADP ratios. The IB strength of sucrose-ADP particleboards was approximately five times greater than binderless board and ADP particleboard (0:100) in the previous study (Komariah *et al.* 2019). However, it was still inferior to the IB strength of PF and pMDI particleboards. Mechanical properties of all the sucrose-ADP particleboards satisfied the type 13 JIS A 5908 standard (2003). Judging from these results, it was observed that the addition of sucrose effectively improved the mechanical properties of OPT particleboard.

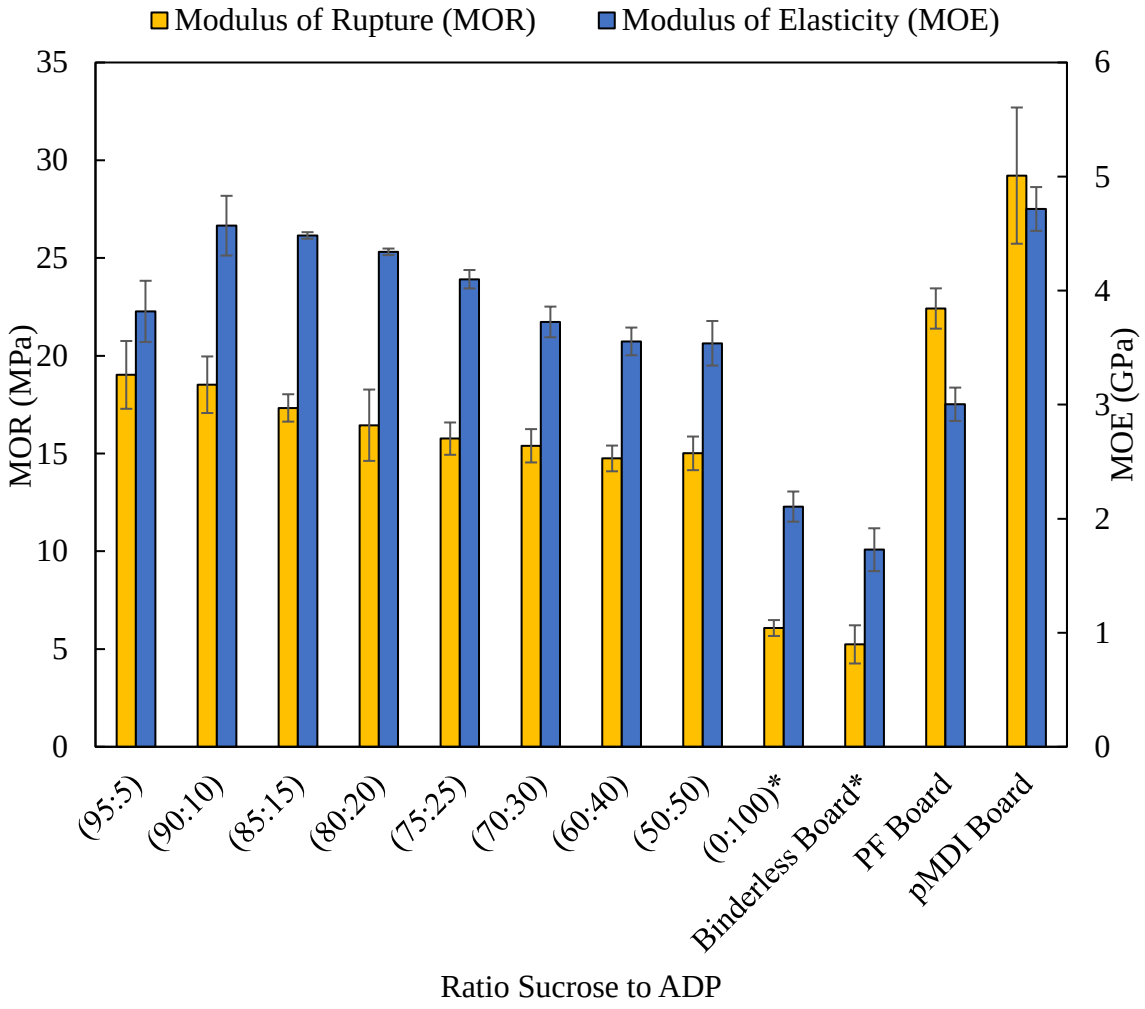



**Fig. 4.1.** Bending properties of the particleboards on various ratio of sucrose-ADP.

*data from Komariah *et al.* (2019)

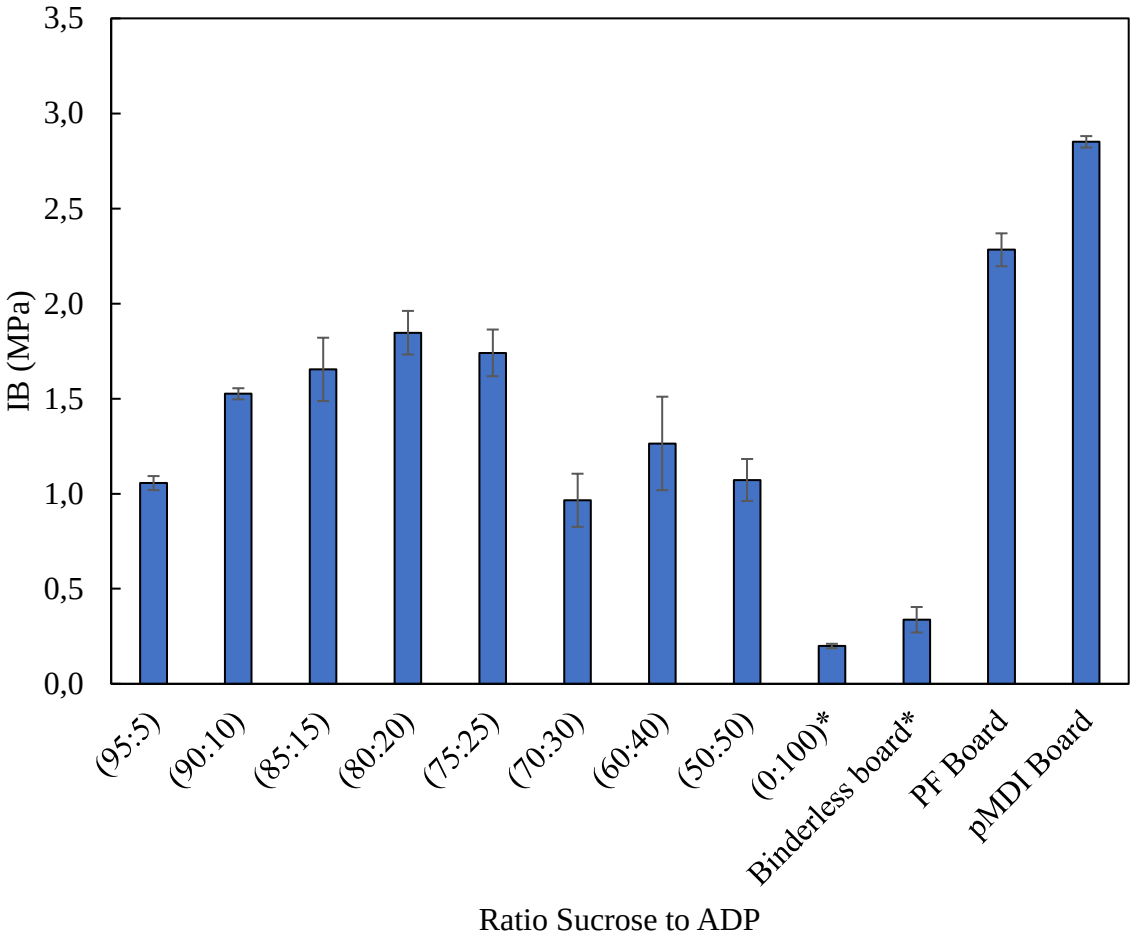



**Fig. 4.2.** Internal bond (IB) properties of the particleboards on various ratio of sucrose-ADP.

*data from Komariah *et al.* (2019)

Figure 4.3 shows the TS and WA values of particleboards made with adhesive of various ratios of sucrose to ADP. The TS value of sucrose-ADP particleboards was in the range of 6.38-13.62%, with the lowest value recorded for the (60:40) sucrose-ADP particleboard. Statistically, the TS value of sucrose-ADP particleboards did not

significantly change ($p>0.05$) between the mixture ratios of 90:10 to 50:50 sucrose-ADP, except for the ratios of (75:25) and (60:40), which were significantly lower than some of the others ($p>0.05$). The TS value of the sucrose-ADP particleboards satisfied the type 13 requirement of JIS A 5908 (2003) (<12%), except for the (95:5) sucrose-ADP particleboard; it was also superior to that of the binderless board and similar to particleboards bonded with PF and pMDI. The WA value of the sucrose-ADP particleboards was in the range of 29.98-41.26%. This was similar to the WA value of PF and pMDI particleboards, but lower than the binderless board. This result indicates that ADP addition ratios from 10-50 wt% maintain the excellent water resistance level of sucrose-ADP particleboards.

Acidity causes the material to seem brittle, hence improving the stiffnes of particleboard and decreasing the strength of particleboard (Kusumah *et al.* 2017). To verify the acidity of the particleboards, the pH values of the soak water from the TS tests were determined. As shown in Fig. 4.4, increases in the ADP content resulted in decreasing pH values of soak water. This means that the acidity of the particleboard increased with each addition of ADP. It is known that ADP decomposes into ammonia, phosphoric acid, diammonium dihydrogen diphosphate and water (Abdel-kadeer *et al.* 1991; Pardo *et al.* 2017) at around 200 °C. Therefore, the decrease of the pH value

would be due to the formation of phosphoric acid. This also could be considered one of evidence for explaining the worsening of mechanical properties with increasing ADP content.

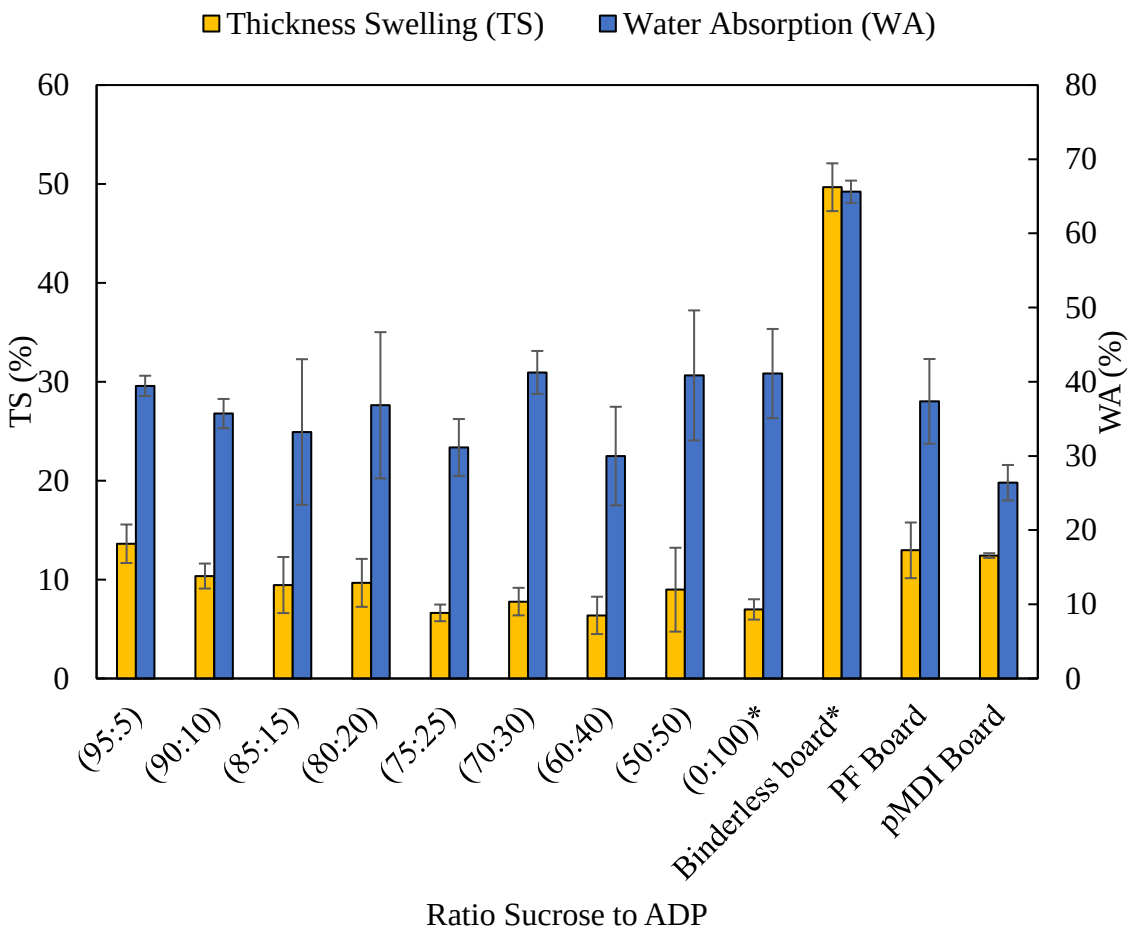



**Fig. 4.3.** Thickness swelling and water absorption of the particleboards on various ratio of sucrose-ADP.

*data from Komariah *et al.* (2019)

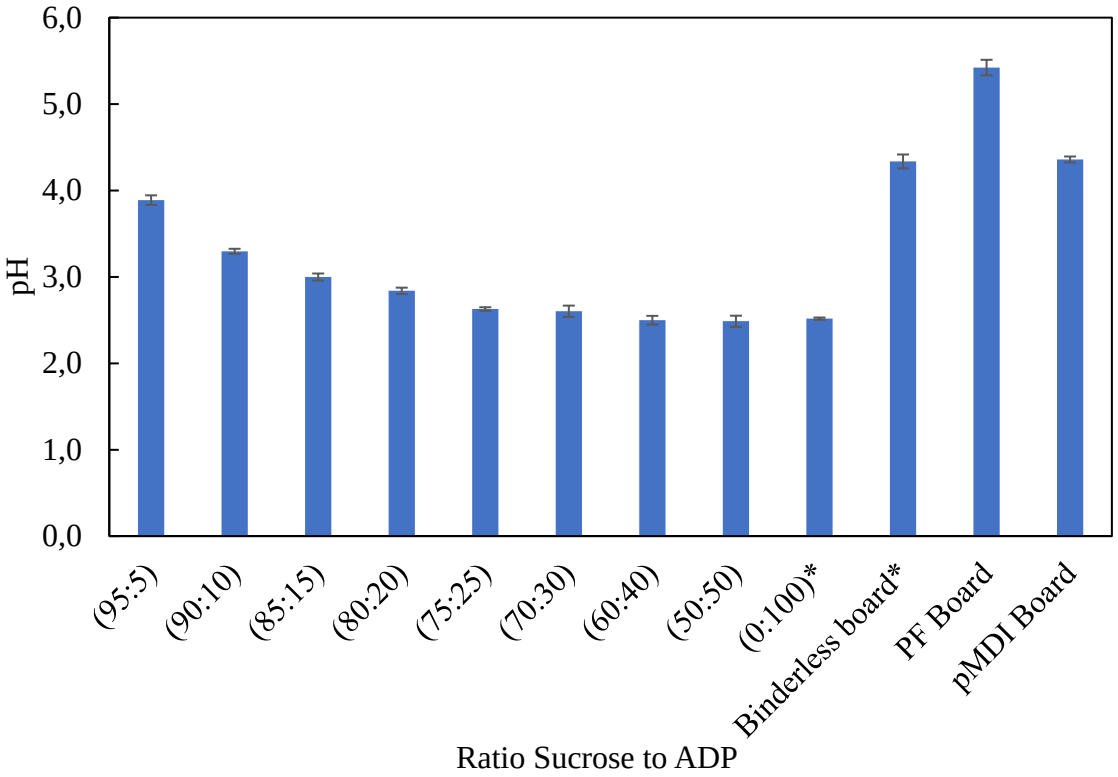



**Fig. 4.4.** pH of soaking water after TS treatment of the particleboards on various ratio of sucrose-ADP.

*data from Komariah *et al.* (2019)

The thickness and weight changes of particleboards were measured continuously by the cyclic aging treatment test to clarify the water resistance under severe conditions in greater detail. Based on the results shown in Fig. 4.3, particleboards with sucrose-ADP ratios of 95:5, 80:20, and 50:50 were used as representative specimens of particleboard to investigate the thickness and weight changes in response to water exposure. The thickness and weight changes of the PF and pMDI particleboards were also measured as

references. The results of thickness and weight changes of cyclic aging treatment are shown in Fig. 4.5. In the thickness changes, the extent of thickness changes from immersion at 20 °C to immersion at 70 °C were relatively higher compared to the change from immersion at 70 °C to that at the boiling point. Moreover, the change at each stage of the cyclic aging treatment decreased with the ADP content increase. The thickness changes of sucrose-ADP particleboards at ratios of 80:20 and 50:50 were less than the thickness changes of sucrose-ADP particleboards at a 95:5, and were similar to the thickness changes of PF board. However, the pMDI board showed a remarkable increase in thickness change after hot-water immersion (70 °C). This indicates that sucrose-ADP particleboards exhibited excellent water resistance, comparable to synthetic adhesives. Remarkably, sucrose-ADP particleboard (80:20) inhibits thickness swelling better than the pMDI board.

The weight changes of sucrose-ADP particleboards were similar at each stage regardless of the ADP content, with final dried weight changes of around -20.38% to -22.58%. Besides, pMDI particleboard showed the lowest final dried weight, around -12.33%, and the lowest weight changes similarly to sucrose-ADP particleboard (80:20). Moreover, PF particleboard showed the greatest weight changes during the immersion and drying treatment, with the final weight change recorded at -27.83%.

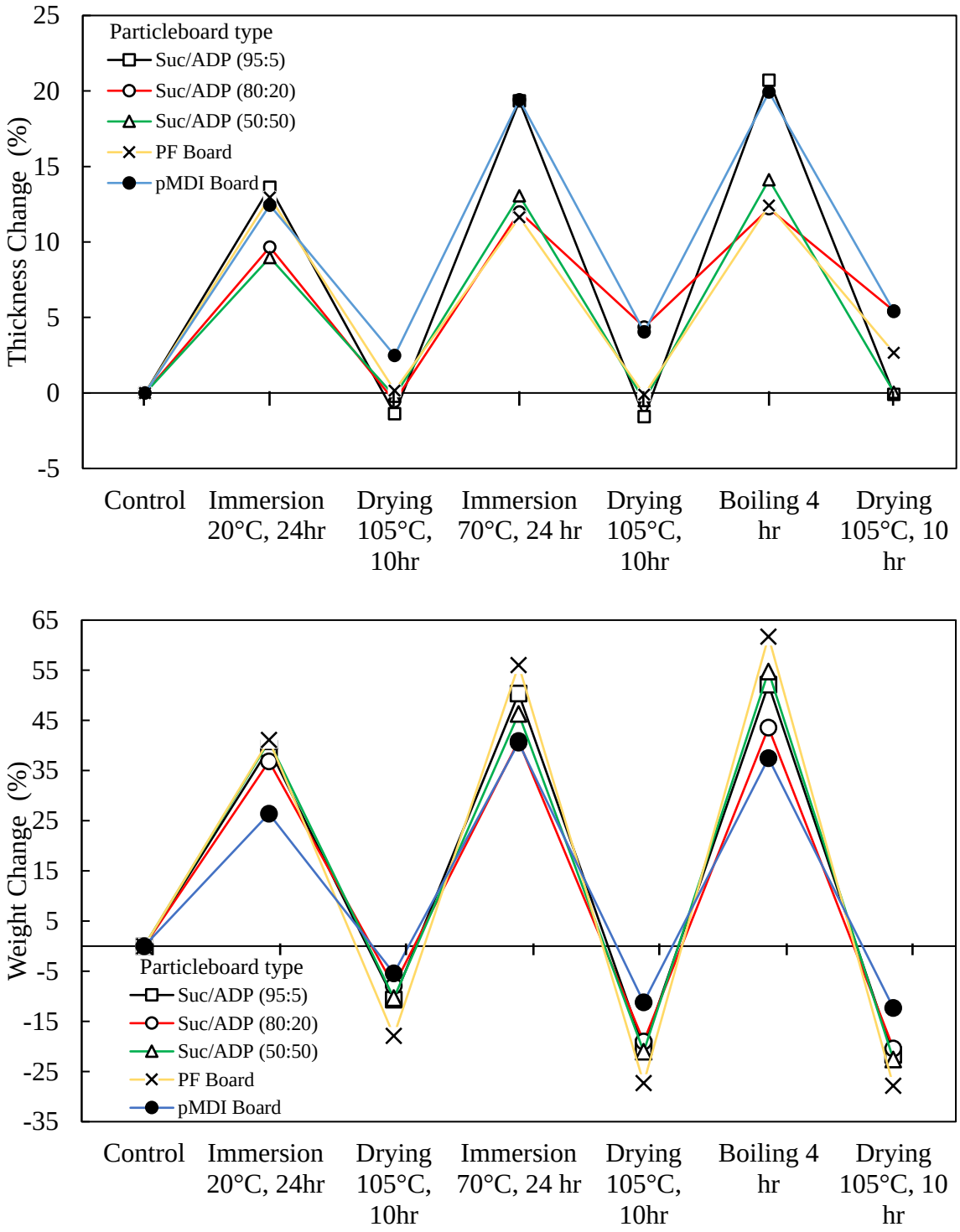



**Fig. 4.5.** Thickness and weight changes of the particleboards on various ratio of sucrose-ADP on the cyclic aging treatment test.

From the comprehensive results of the tests of mixture ratios on the particleboard properties, it could be concluded that the sucrose-ADP ratio of 80:20 was the most effective for particleboard, with relatively high MOR, MOE, and lower TS values compared to other mixture ratios. Therefore, this adhesive condition was utilized in the second part of this study.

### 4.3.2. Effects of resin content

The effects of resin content on the mechanical and physical properties of OPT particleboard were investigated using sucrose-ADP adhesive at the ratio of 80:20. Figure 4.6 shows the bending properties of the sucrose-ADP-adhesive OPT particleboards with various resin contents (wt%). The MOR values gradually increased with increasing resin content, while the MOE values tended to increase with increasing resin content up to 20 wt%, then slightly decreased thereafter. Statistical analysis showed no significant difference ($p>0.05$) among MOR values of particleboards with resin contents of 10 to 30 wt%; however, MOE values for resin content of 20 wt% were significantly higher ($p>0.05$) than MOE values of particleboards with other resin contents. This means that 20 wt% of resin content is effective in obtaining good bending properties. All tested particleboards satisfied the bending strength type 13 JIS A 5908 standard (2003). This is

in line with Lamaming *et al.* (2014), who found that the addition of sucrose at rates as high as 20 wt% improved the properties of OPT binderless board. Moreover, Stark *et al.* (2010) noted that resin content (4-15 wt%) is needed for synthetic adhesive particleboard. Then, a much resin content for sucrose-ADP particleboard was needed to obtain satisfactory properties.

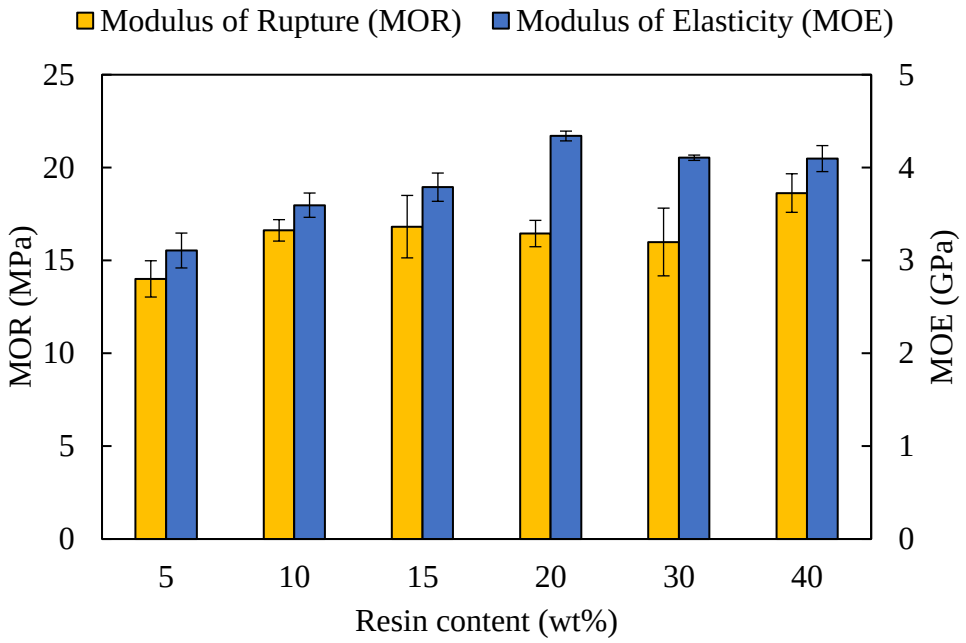



**Fig. 4.6.** Bending properties of the particleboards with sucrose-ADP 80:20 on various resin content (wt%).

The IB strengths of the tested particleboards are shown in Fig. 4.7. The results showed that the IB strength has a similar trend to the bending strength, with the IB value gradually increasing with resin content, reaching a maximum at 20 wt% resin content, and then decreasing. The average value at 20 wt% was 1.84 MPa, almost two times higher

than that at 5 wt%. Compared to the increase of the bending properties, a remarkable increase was recognized. The IB strength required in the 13 types of JIS A 5908 is more than 0.2 MPa. Therefore, the adhesion system composed of sucrose and ADP developed a good dry-bond strength.

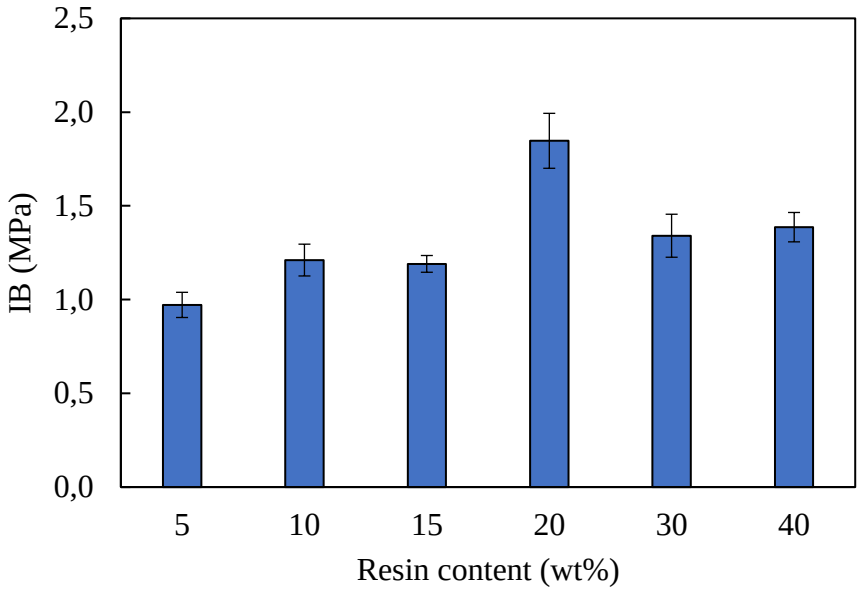



**Fig. 4.7.** Internal bond (IB) properties of the particleboards with sucrose-ADP 80:20 on various resin content (wt%).

Figure 4.8 shows the TS and WA values of the particleboards with various resin contents (wt%). The results show trends of decreasing TS and WA values with the increasing resin content. The TS value was about 12% or less when resin content was 20 wt% or more. Generally, the main factors affecting TS are the compressibility of the board, the adhesive type, and the resin content. In this study, the target density of the

particleboard was constant, irrespective of the resin content. This means that an increase in the resin content leads to a decrease in the number of particles. The decrease of the relation-force of compressed particles and the coating of particles with the adhesive resulted in decreased TS. Furthermore, the pH values of the soaking water from the TS test are shown in Fig. 4.9. Increases in the resin content resulted in decreasing soak-water pH; in other words, the more adhesive, the more acid formation.

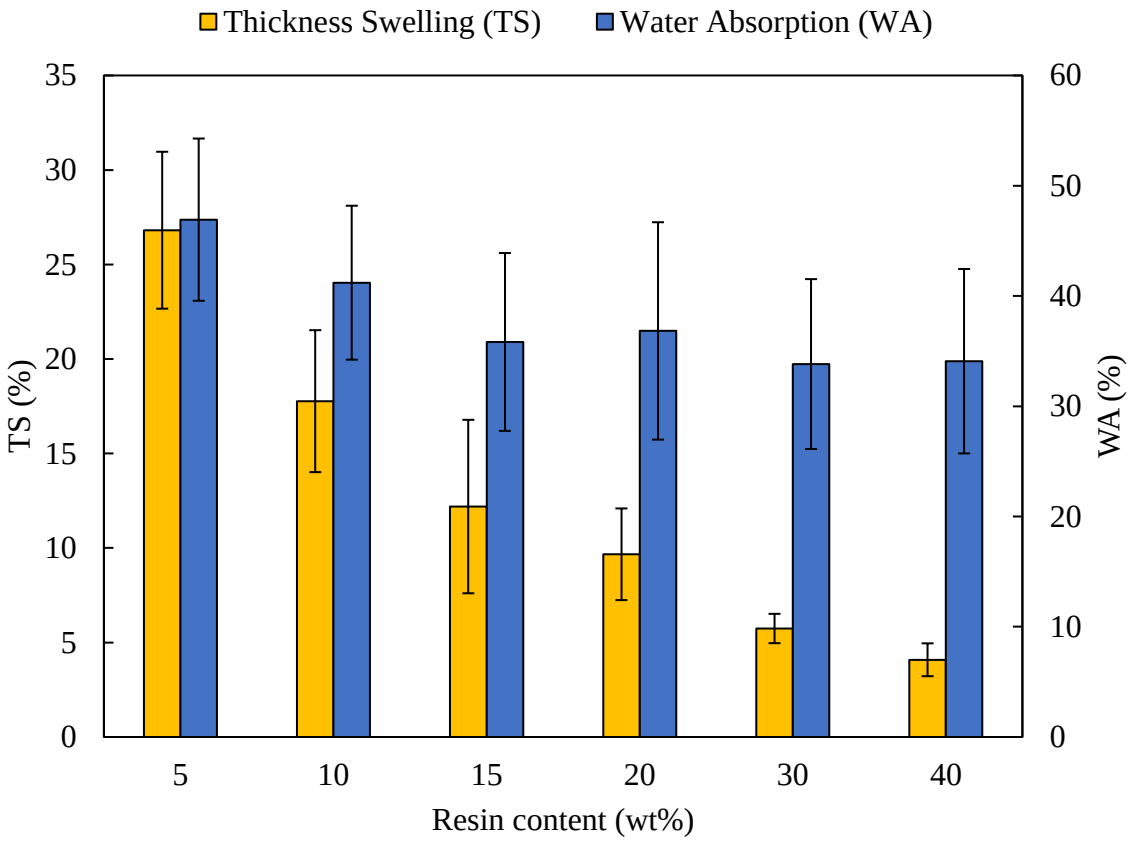



**Fig. 4.8.** Thickness swelling and water absorption of the particleboards with sucrose-ADP 80:20 on various resin content (wt%).

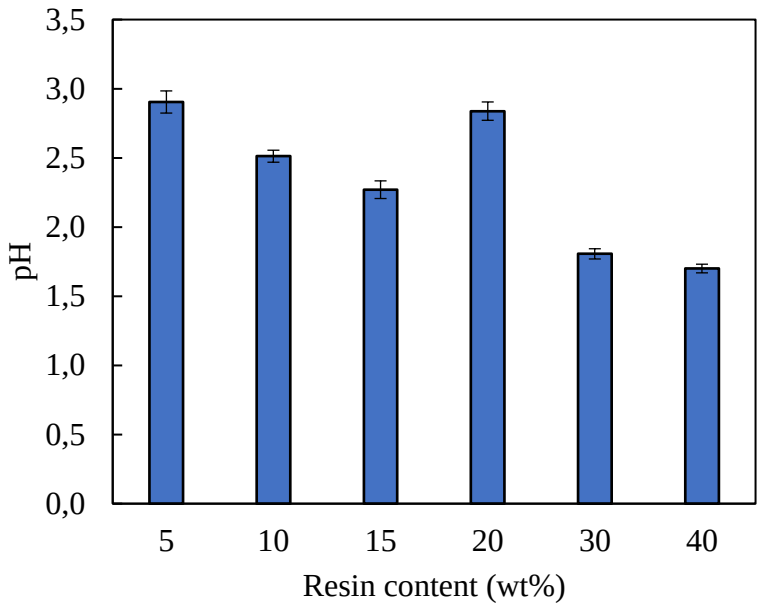
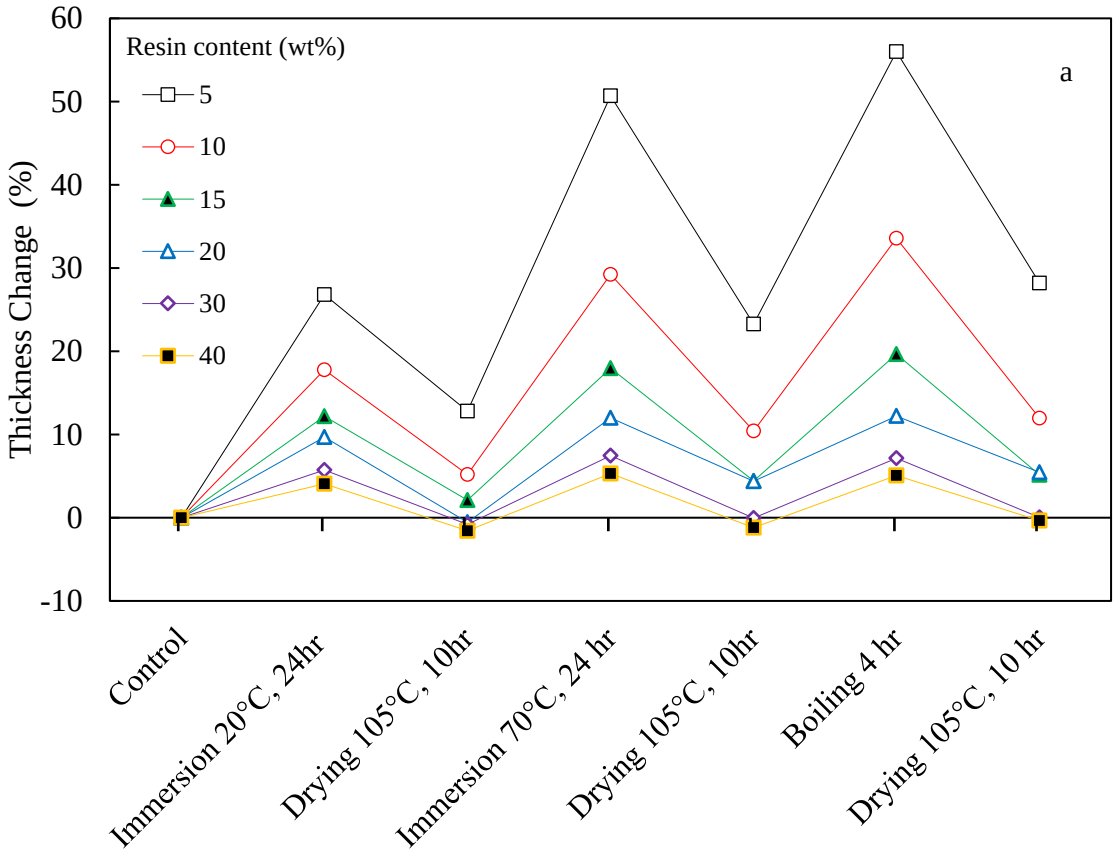



**Fig. 4.9.** pH of soaking water after TS treatment of the particleboards with sucrose-ADP 80:20 on various resin content (wt%).

The thickness and weight changes of particleboards with different resin contents (wt%) in a cyclic aging treatment are shown in Fig. 4.10. In the cyclic aging treatments, the thickness of all types of particleboards increased in each immersion treatment. The degree of thickness change decreased with increasing resin content and was remarkably suppressed when 20 wt% and higher resin contents were applied. The final dried thickness changes of sucrose-ADP particleboards with resin content 20-40 wt% were between -0.35% and 5.44%. Furthermore, the weight changes in the first water immersion treatment exhibited decreasing values as the resin content increased. This indicated that increasing resin content inhibits water absorption. The overall weight increase in the subsequent

warm water immersion treatment was higher than that in the first treatment. This would be due to the lowering of the water resistance of the adhesive and the penetration of water into the OPT. However, no marked change in weight with the subsequent boiling water treatment was observed, and the final dried weight changes recorded were between -17.34% and -20.12%. The weight changes of particleboards with resin content of 20 and 30 wt% were remarkably smaller than those of other conditions.



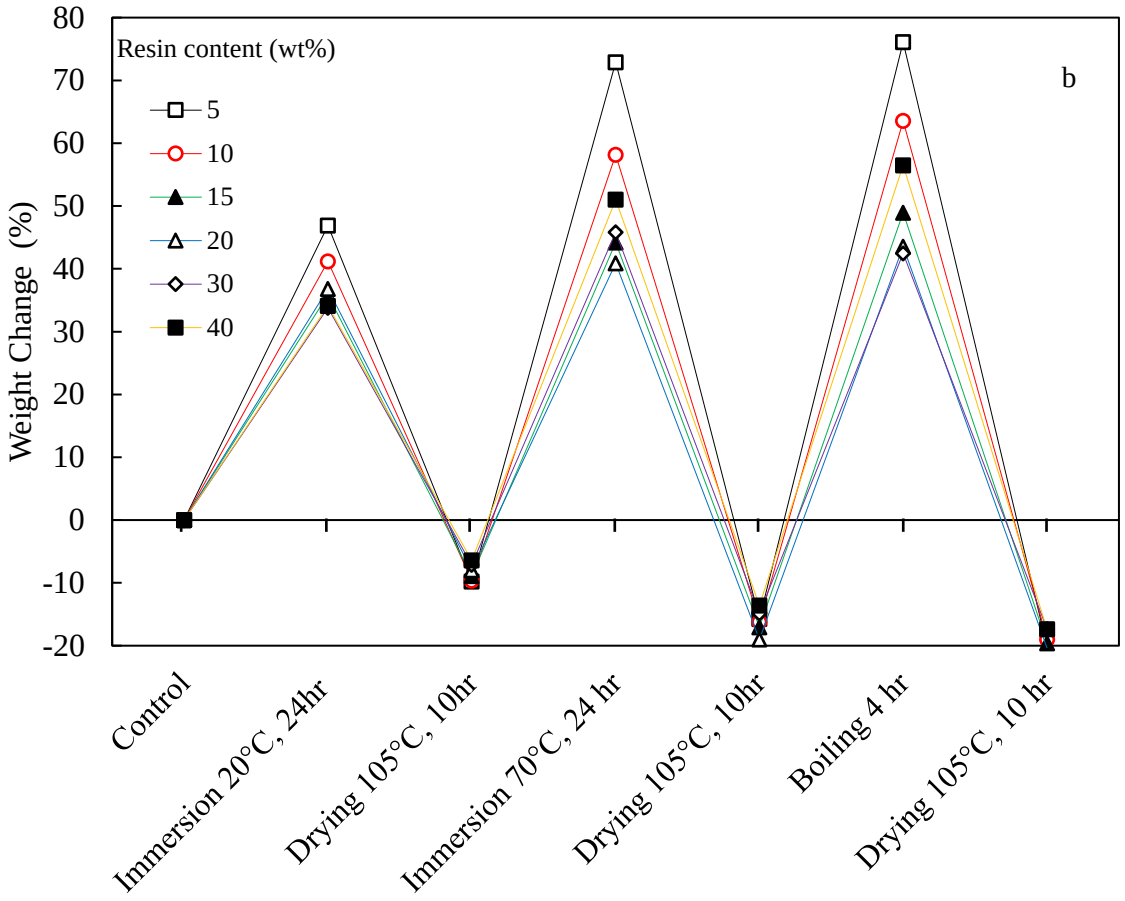



**Fig. 4.10.** Thickness (a) and weight (b) changes of the particleboards with sucrose-ADP 80:20 on various resin content (wt%) on the cyclic aging treatment test.

### 4.3.3. Termite and decay resistance

The biological durability against termites and decay of sucrose-ADP (80:20) and resin content of 20 wt% particleboard was investigated. The biological durability of binderless board, ADP, PF, and pMDI particleboards were also investigated as references. Table 4.3 shows the average mass loss and mortality in the particleboards exposed to termite attack. Particleboard bonded with sucrose-ADP showed a mass loss and termite

mortality of 13.61% and 19.55%, respectively. These values were similar to the mass loss and termite mortality of ADP and PF particleboards. Meanwhile, pMDI particleboard showed lower mass loss and termite mortality than sucrose-ADP particleboard, and the binderless board showed similar mass loss but lower termite mortality compared to the sucrose-ADP particleboard. This indicated that chemicals derived from sucrose and ADP might have inhibitory properties against termites.

**Table 4.3.** Termite and decay resistance of OPT binderless board and OPT particleboard bonded with sucrose, ADP, PF and pMDI.

| Specimen Types | Biological Durability | | | |
|---|---|---|---|---|
| | Termite Resistance | | Decay Resistance | |
| | Termite Mortality (%) | Mass loss (%) | Mass Loss of the Brown Rot Fungi (%) | Mass Loss of the White Rot Fungi (%) |
| S-ADP Board | 19.55 (3.58) | 13.61 (1.44) | 16.80 (1.28) | 16.31 (0.83) |
| ADP Board | 21.82 (3.78) | 12.53 (0.54) | 21.61 (0.89) | 16.67 (0.33) |
| Binderless board | 3.03 (0.43) | 14.35 (2.62) | 63.93 (5.77) | 76.15 (8.27) |
| PF board | 15.39 (4.45) | 12.97 (0.67) | 26.21 (0.47) | 24.48 (0.41) |
| pMDI board | 8.61 (0.66) | 9.89 (0.63) | 13.47 (1.83) | 12.42 (0.29) |
| Sugi wood (Control) | 3.27 (1.64) | 25.80 (3.08) | 58.46 (3.99) | 22.20 (6.87) |

*S-ADP (80:20) Resin Content 20 wt%

The mass losses of particleboards exposed to fungal decay is also presented in Table 4.3. The mass loss values for white-rot fungi and brown-rot fungi on the sucrose-ADP particleboard were similar, around 16%. The pMDI particleboard showed the lowest mass loss values for the white-rot fungi and brown-rot fungi, 13% and 12%, respectively while binderless particleboard showed the highest mass losses, 63% and 76%, respectively. Compared to the PF particleboard, sucrose-ADP particleboard caused a lower mass loss in the fungal decay test. These results suggest that the sucrose-ADP adhesive system in the particleboard tested in this study protects against fungal decay. In a previous study, Kusumah *et al.* (2017) found that sorghum bagasse particleboard with sucrose-citric acid adhesive at weight ratios of 85/15 and 90/10 had good termite resistance and effectively inhibited decay. It can be concluded that the sucrose-ADP particleboard has good biological durability, similar to that of particleboards made with synthetic adhesives.

### 4.3.4. FTIR analysis

The infrared (IR) spectrum of particleboard with a sucrose-ADP ratio of 80:20 and resin content of 20 wt% was taken to clarify the chemical changes of OPT particleboard. Figure 4.11 shows the results of IR spectra of binderless board, ADP

particleboard and sucrose-ADP particleboard after cyclic aging treatment. Two absorption peaks at approximately 1728 and 1510 $cm^{-1}$ were observed in all specimens. The peak at 1728 $cm^{-1}$ is typically ascribed to the stretching vibrations of carbonyl C=O bonds (Lee *et al.* 2018), which are formed upon heating of sucrose. Peaks at 1510 $cm^{-1}$ have been attributed to both the C=C of the aromatic ring vibrations in lignin (Sun *et al.* 1999) and the furan ring from sucrose heated with ADP (Umemura *et al.* 2017 and Komariah *et al.* 2021). Therefore, the peak at around 1510 $cm^{-1}$ likely combines the vibrations of two different functional groups. These two peaks (at 1728 and 1510 $cm^{-1}$) in the sucrose-ADP particleboard spectra were higher than those of binderless board and ADP particleboard. This indicates that the formation of the furan ring and carbonyl group was significant in the sucrose-ADP particleboard. This would be reasonable because their formation derives from both sucrose and the saccharides of OPT. The addition of a furan ring contributes to the enhancement of bondability. Consequently, the mechanical and physical properties of the particleboard were improved by adding sucrose. Zhao *et. al.* (2019a) mentioned that the main reactions of curing process of the sucrose-ADP adhesive system is the dehydration condensation of furan compounds and the Maillard reaction, with the resulting substance is mainly polyfuran linked by C-O-C and C=N-C bonds. However, the formation of C-O-C and C=N-C bonds were not found in this study.

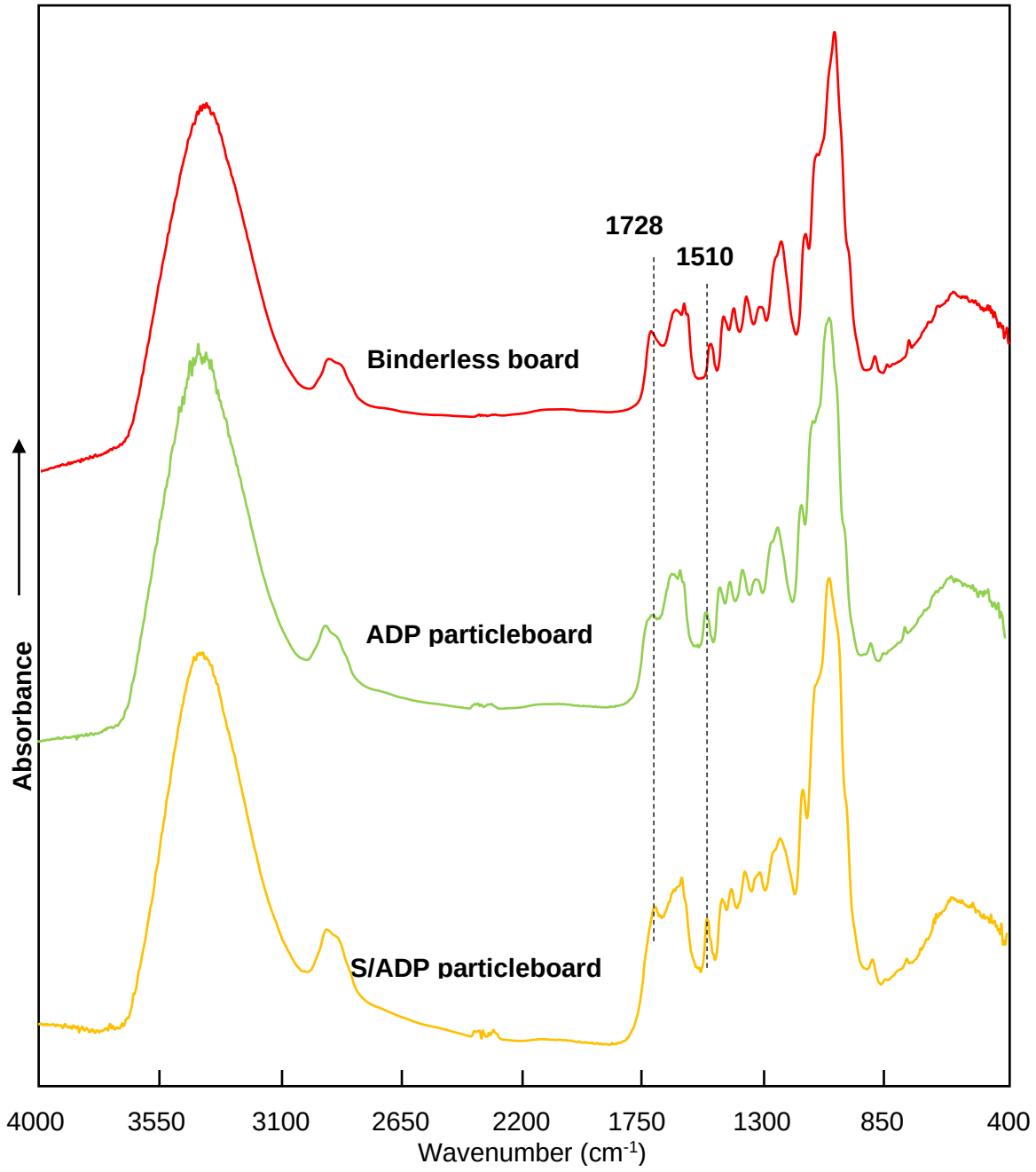



**Fig. 4.11.** FTIR spectra of OPT binderless board, ADP particleboard, and S/ADP (80:20 RC 20 wt%) after cyclic aging treatment test.

## 4.4. Summary

The effects of sucrose-ADP adhesive on the properties of particleboard made from the inner part of OPT were investigated. It was clarified that the mechanical properties and water resistance increased as the sucrose ratio increased. In this study, the optimum sucrose-ADP mixture ratio and the optimum resin content was (80:20) and 20 wt%, respectively. The MOR, MOE, and TS values were 16.45 MPa. 4.34 GPa, and 9.67%, respectively. The IB strength was 1.85 MPa, and the board exhibited good bond performance. The particleboard manufactured under the optimum condition was comparable to the 13 types of JIS A 5908. The particleboard also had excellent water resistance under cyclic aging treatment. In addition, the sucrose-ADP adhesive in the particleboard provided good protection against termite and decay, similar to that of particleboards with synthetic adhesives. Therefore, excellent particleboard could be manufactured from the inner part of OPT and a sucrose-ADP adhesive.

# Conclusions

Generally, this study purposed to develop good physical and mechanical properties of particleboard made from inner part of oil palm trunk (OPT) using methods that do not depend on synthetic resin adhesives from fossil resources. For this objective, the particleboards were manufactured by utilization of chemical components of inner part of OPT, ammonium dihydrogen phosphate (ADP) and sucrose. The details were discussed in the following chapters.

In Chapter 1, the development of agro-based particleboard and its characteristics that suitable for production particleboard in recent research were discussed. Oil palm as one of the agricultural plants that has the potential to use as raw material for particleboards, also discussed its current situation. Specifically, characteristics of lignocellulosic materials from oil palm trunks were discussed due to their abundant availability. Generally, the particleboard is bonded with synthetic resins that contain harmful chemical agents from unsustainable resources i.e. petroleum fossil resources. Even though there are researchers that attempted to manufacture binderless boards without using fossil resources-based adhesives. However, the performance of binderless boards is generally very poor compared to boards that use a suitable synthetic resin adhesive, especially

water-resistant properties. Based on the recent condition of natural-based adhesive development in manufacturing of wood particleboard, ADP and sucrose had high prospective to be used as environmentally friendly natural-based adhesive. According to the background in this study, the objective of this study is to develop good physical and mechanical properties of particleboard made from inner part of OPT using methods that do not depend on synthetic resin adhesives.

In Chapter 2, the effects of adding ADP on the characteristics of particleboard manufactured using the inner part of OPT were investigated. Particleboards were manufactured based on the results of the thermal analyses of OPT particles added ADP. The particleboards were manufactured under the pressing conditions of 180 °C for 10 min. The ADP content was varied in the range of 0 to 40 wt%. The mechanical and physical properties were improved with the addition of ADP until 10 wt%. However, the addition of ADP 20 to 40 wt% brought a decline in the bending and internal bonding properties, due to an excess of ADP in the particleboard. The mechanical and physical properties of particleboard with 10 wt% ADP addition satisfied the requirement of the JIS A 5908 (2003) 8 type. Particularly, the particleboards made with addition of ADP more than 10 wt% had excellent water resistance. Based on the FTIR results, a change of the carbonyl group was observed, and the existence of ADP as a water-soluble component was

recognized. According to the result of cyclic aging treatment, it was suggested that water-soluble components of OPT changed to water-insoluble components by the addition of ADP. Consequently, the addition of ADP until 10 wt% was effective for improving the physical and mechanical properties of particleboard using the inner part of OPT.

Therefore, in Chapter 3, the ADP's influences on the physical and chemical changes of a water-soluble extractive of the inner part of OPT were clarified. The extractive was mixed with various ADP solid contents, i.e., 0, 10, and 40 wt%. With heating, the extractive changed into an insoluble substance, and the addition of 10 wt% ADP doubled the insoluble substance amount. Based on the FTIR results, the formation of furan compounds and carbonyl group derived from sugars was inferred. And the thermal analysis showed that the formed insoluble substance had good thermal stability, suggesting a high-molecular-weight substance. Then, the effect of the water-soluble extractive on binderless particleboard with the addition of ADP was also investigated. Particleboards using hot-water-treated particles were manufactured. The manufacturing condition was similar methods that was described in Chapter 2. The boards' physical and mechanical properties irrespective of ADP addition tended to be inferior to those of particleboards in Chapter 2 (without extracted treatment). In particular, the water-soluble extractive was essential for the water resistance of the particleboard. Considering these

results, the saccharides in water-soluble extractive have an important role in the properties of particleboards made from the inner part of OPT. Therefore, in Chapter 4, the improvement of mechanical and physical properties of OPT particleboards were observed by adding sucrose to adhesion system.

In chapter 4, the effect of adding sucrose with ADP as an adhesive on the improvement of physical and mechanical properties of particleboard using the inner part of OPT were expected. The effects of different ratios of sucrose and ADP and different resin contents were investigated. Moreover, particleboards manufactured using phenol formaldehyde (PF) resin and polymeric methylenediphenyl isocyanate (pMDI) were used as references. It was clarified that the mechanical properties and water resistance increased as the sucrose ratio increased. In this study, the optimum sucrose-ADP mixture ratio and the optimum resin content was (80:20) and 20 wt%, respectively. The particleboard manufactured under the optimum condition was comparable to the 13 types of JIS A 5908. The particleboard also had excellent water resistance under cyclic aging treatment, and comparable to PF and pMDI particleboards. Remarkably, sucrose-ADP particleboard (80:20) inhibits thickness swelling better than the pMDI board. In addition, the sucrose-ADP adhesive in the particleboard provided good protection against termite and decay, similar to that of particleboards with PF and pMDI. According to the results

of infrared spectra measurement, the furan ring and carbonyl group was generated by the reaction between sucrose, ADP, and the saccharides of OPT. Therefore, excellent particleboard could be manufactured from the inner part of OPT and a sucrose-ADP adhesive.

In brief, development of particleboard using the inner part of OPT had some benefit values of environmental and technical aspects. Utilization of the inner part of OPT particles in the manufacturing of particleboard could utilize waste from oil palm plantation into some added-value products also reserve the carbon for long term. In addition, sucrose and ADP as sustainable natural resources has well prospective to substitute synthetic adhesive from fossil resources for wood-based composite adhesive. Technically, the particleboard had good physical properties especially water-resistant properties. Moreover, the particleboard has good durability against termite and decay, hence the particleboard suitable for interior and exterior uses.

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