# CHAPTER III

# MATERIALS AND METHODS

## 3.1 Materials

### 3.1.1 Source of oil palm trunk

Thirty oil palm trunks (OPT) were provided by MPOB (Malaysian Palm Oil Board). The oil palm trees of high yielding *tenera* variety (a hybrid between those of *dura* and *pisifera*) were collected during the replanting operations at Sungei Kahang Estate in Johor. Prior to felling, only those oil palm trees whose bole is straight and free from visible defects were selected. All sample trees were classified under the same diameter class between 45 to 52 cm (measured at one meter above the ground) and the bole length ranged from 11 to 13 m long. After measuring the length (Figure 3.1), the palm trunk was then cross cutting to three samples billets, each of 4 m long.

Figure 3.1: Length of felled oil palm trunk prior to cross cutting into billets

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The billets were labelled with codes indicating the palm number and its position within the tree height. The billets were loaded onto a lorry and transported to MPOB/UKM Research Station, which is located in Pekan Bangi Lama, Selangor for the preparation of oil palm lumber samples. The breakdown of OPT billet into lumber scantlings was done using a 9-foot band headrig with a log carriage (Figure 3.2). After securing on the log carriage with dog spikes, the OPT billet was fed into the saw by a powered winch. The saw made a single cut on each pass.

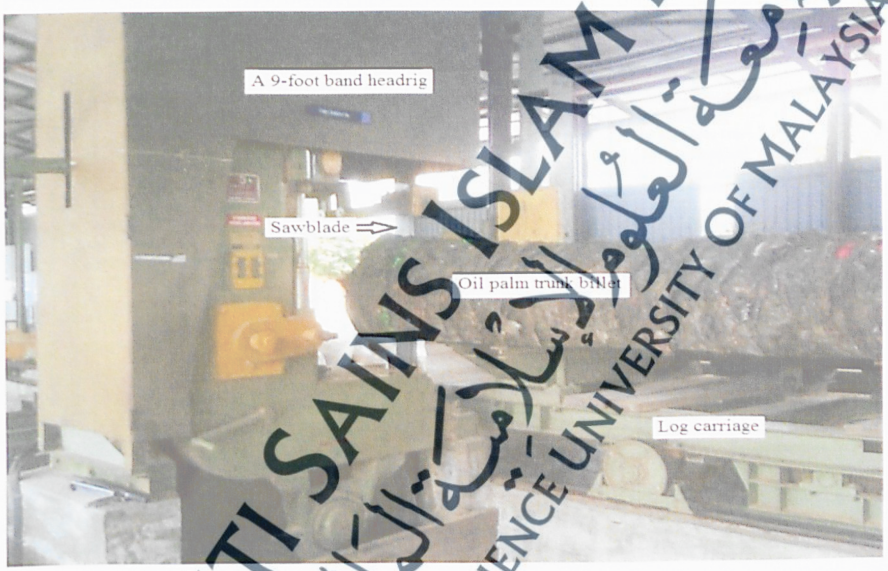


Figure 3.2: Primary breakdown of oil palm trunk into sawn lumber using a 9-foot band headrig with log carriage

The cut material collected from out-feed rollers and the remainder of the log had taken back past the saw and repositioned it to another cut. With a log carriage, the OPT billet was turned between cuts to maximize the quality and the value of log cut. The vertical knees of the log carriage, against which the log was dogged, can move independently to allow for log taper. This allows the sawing of OPT billet to

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its full-length slab, cut parallel to the pith. Feed speed was 1.2 m $s^{-1}$, and the return speed was twice as fast.

## 3.2 Physical Properties of Oil Palm Trunk

### 3.2.1 Preparation of test samples

Five OPT were selected to determine the moisture content and basic density in pith to periphery zone with tree heights, is illustrated in Figure 3.3.

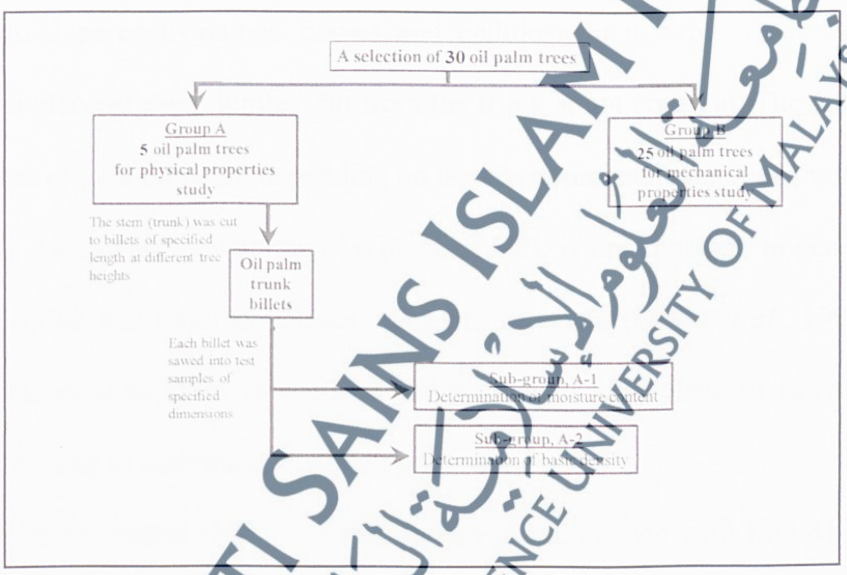


Figure 3.3: Preparation of oil palm lumber for physical properties study

The OPT in group A was divided into two subgroups, namely (a) Subgroup A-1, determination of moisture content, and (c) Subgroup A-2, determination of basic density. After sawing, the test samples were then labelled by using a permanent marker pen with codes according to palm number, height level, position and the location of test sample taken across the diameter. Test samples were marked using the following codes: Inner zone (pith), test sample taken in pith to 50 mm towards

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periphery; Middle zone (intermediate), test sample taken at 50 mm to 100 mm from the pith towards periphery; Outer zone (periphery), test sample at 100 mm to 150 mm from the pith towards east. Procedures for sampling to determine the physical and mechanical properties were done in accordance with International Standards Organisation, ISO 3129-1975 (1975).

### 3.2.2 Determination of moisture contents

The OPT is a porous material containing numerous elements such as air, water, ground parenchymatous tissues and cellulosic materials. As a result, the weight of a piece of sawn lumber cut from the trunk is not constant. The sawn OPL tends to lose or gain moisture depending on the environmental conditions to which it is exposed. Moreover, the volume of a piece of OPL is not constant. In general, the OPL will shrink and swell as it loses and gains moisture (Cown *et al.*, 1996). It is therefore essential to know how much water contains in a piece of sawn lumber before attempting to determine any other property.

Moisture content (MC) was determined in accordance with ISO 3130-1975 (1975) procedures. After sawing, the test samples (in subgroup A-1) having a square cross-section of 20 mm and the length, along the vascular bundles, of 25 ± 5 mm were stored in a plastic bag. This was carried out in order to ensure its moisture content will remain unchanged during storage. Test samples were weighed individually to the nearest 0.01 g to obtain the green weight. After weighing, the samples were dried in an electric oven (Memmert, UFE 600) at 103 ± 2 °C until constant in order to obtain the dry weight to the nearest 0.01 g. The samples were

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further reweighed at equal hourly intervals and the weights recorded until no weight loss was detectible.

At the end of the final period, test samples were cooled in a dessicator over silica gel. After cooling, the sample was weighed rapidly enough in order to avoid an increase in moisture content by more than 1%. The difference in the two values between the initial and constant weights is assumed to be due to loss of water by evaporation during drying. The moisture content, $MC_{od}$ of each test sample, as a percentage by mass were calculated as follows (ISO 3130-1975):

$$MC_{od}\,(\%) = \frac{(W_g - W_{od})}{W_{od}} \times 100 \qquad \text{(Equation 3.1)}$$

where $MC_{od}$ is the moisture content on an oven-dry basis in percentage, $W_g$ is the mass, in grams, of the test sample before drying, and $W_{od}$ is the mass, in grams, of the test sample after drying.

### 3.2.3 Determination of basic density

The density of OPT is measured once its MC has been defined. In physics, the density of a material is described as the mass per unit volume (kg $m^{-3}$). The situation is quite complicated because changes in moisture will affect both the mass and volume (Olesen, 1971). The term 'basic' emphasizes that both parameters measured, the oven-dry mass and the swollen volume, have constant and reproducible values. Hence basic density would be the most useful descriptor of OPT density.

The BD was obtained using green volume and oven dry mass of each test sample in accordance with ISO 3131-1975 procedures. Test samples (in subgroup A-2) were prepared in the form of a square cross-section of side 20 mm and length



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along the vascular bundles of 25 ± 5 mm. In order to ensure the moisture content remains unchanged, the test samples were stored in a plastic bag after sawing.

The weight of test samples was measured with a digital balance to the nearest 0.01g while its volume to the nearest 0.01 ml was determined by the water immersion method before drying. The samples were coated with molten wax and immersed in water. The weight and the latest water level of volumetric cylinder were recorded. After soaking, the specimens were oven dried in an electric oven at 103 ± 2°C until constant weight to obtain the dry weight to the nearest 0.01 g, and cooled in a dessicator over silica gel. After cooling in a dessicator over silica gel, the test sample was weighed rapidly enough in order to avoid an increase in moisture content by more than 1%.

The basic density, BD of each test sample, as a ratio of mass per unit volume was calculated from the relationship as follows (ISO 3131-1975):

$$BD\ (kg\ m^{-3}) = \frac{W_{od}}{V_g} \qquad \text{(Equation 3.2)}$$

where BD is the basic density in kg $m^{-3}$, $W_{od}$ is the oven dried mass of test samples in kg, $V_g$ is the volume of green test specimen in $m^3$.

## 3.3 Mechanical Properties of Oil Palm Lumber

### 3.3.1 Preparation of oil palm lumber samples

A total of 25 OPT were selected to obtain lumber scantlings for this study, is given in Figure 3.4. Oil palm billets of 80-cm long were cut at the 2-m, 4-m and 6-m height levels. This technique should sample the OPT, taken as a whole. From group B, the oil palm billets were divided into two subgroups as follows: (a) Subgroup B-1, determination of the bending strength of matched OPL samples



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dried at 10 ± 2% moisture content, and (c) Subgroup B-2, determination of the bending strength of matched dry OPL samples subjected to a gum rosin and densification treatments.

The billets of each subgroup B-1 and B-2 were then sawed to produce lumber scantlings with nominal dimension of 100 mm wide in radial by 100 mm thick in tangential and 800 mm long in longitudinal directions. After sawing, the lumber scantlings were then labelled by using a permanent marker pen with codes indicating the position and the location within the bole diameter with tree heights.

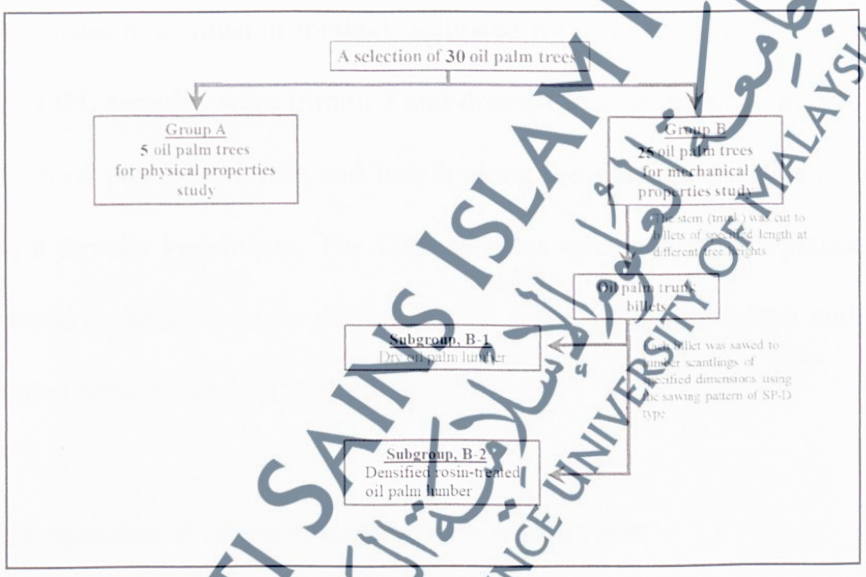


Figure 3.4: Preparation of oil palm lumber for mechanical properties study

### 3.3.2 Treatments of oil palm lumber

#### 3.3.2.1 Dry sawn lumber

For subgroup B-1, the OPL samples were left to dry in an electric oven at 60 ± 2 °C and weighed daily until the MC reached 10 ± 2%. After drying, the OPL samples were trimmed and dressed to size in the form of a square cross-section of side 20 ± 1 mm and length along the vascular bundles of 300 ± 1 mm using a



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circular bench saw. The OPL samples were stored in a plastic bag in order to avoid moisture changes during storage. The OPL dimensions and its MC were recorded prior to bending strength tests.

#### 3.3.2.2 Densified gum rosin-treated lumber

From subgroup B-2, the OPL samples were left to dry in an electric oven at 60 ± 2 °C and weighed daily until the moisture content reached 10 ± 2% in order to obtain its matched dried samples. Dry OPL samples were then treated with a gum rosin using a vacuum infusion method, followed by a densification process. After curing, the OPL samples were trimmed and dressed to size in the form of a square cross-section of side 20 ± 1 mm and length along the vascular bundles of 300 ± 1 mm using a circular bench saw. The OPL samples were stored in a plastic bag in order to avoid moisture changes during storage. The OPL dimensions and its MC were recorded prior to bending strength tests.

### 3.3.3 Impregnation of oil palm lumber with a gum rosin

#### 3.3.3.1 Gum rosin

Gum rosin, which is in the form of crystal was purchased from Acros Organics (M) Sdn Berhad. Being a natural organic compound, the gum rosin was dissolved with methyl ethyl ketone (MEK) (Phillips *et al.*, 1995; Erwinsyah, 2008). Table 3.1 gives the general information pertaining to the gum rosin used.

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Table 3.1: Properties of gum rosin used

| Name | Concentration (%) | Viscosity (cP) | Temperature ($^0$C) |
|---|---|---|---|
| Rosin gum | 60 | 180 ~ 210 | 28.2 |

### 3.3.3.2 Vacuum infusion process

In this study, the basic principles of the VI process used were similar to the process developed and the method outlined by Kamarudin and coworkers (2007). In general the impregnation of gum rosin was based on equipment and method that uses pressure gradient to drive gum rosin into the OPL matrix (Figure 3.5).

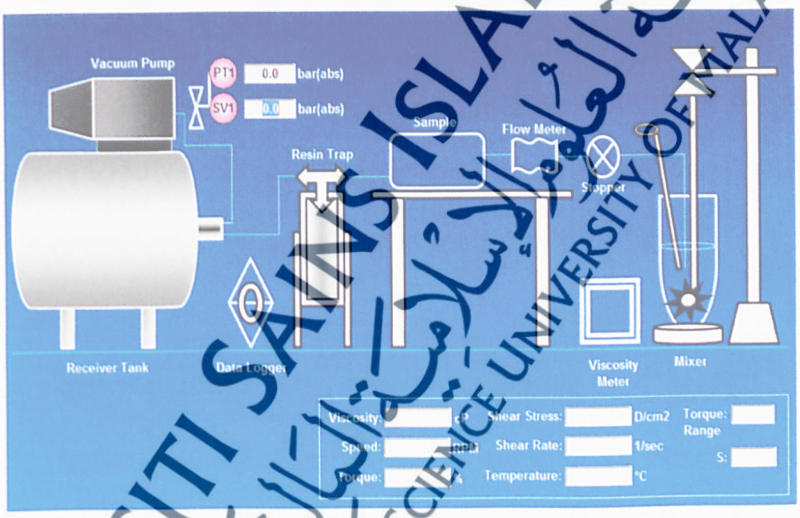


Figure 3.5: Flow process of the major stages for impregnation of oil palm lumber with gum rosin using the vacuum infusion system

To impregnate the OPL, dry lumber sample was laid in a flexible and airtight bag with certain positions being opened for gum rosin supply and outlets (Figure 3.6). Prior to infusion, the entire seals and pleats within the system was checked for leaks in order to avoid the formation of voids. This is because the process is sensitive

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to leakage in the flexible membrane, of which the smallest amount of air being introduced would probably result in gum rosin pooling, under-saturation, or a complete stoppage of gum rosin flow.

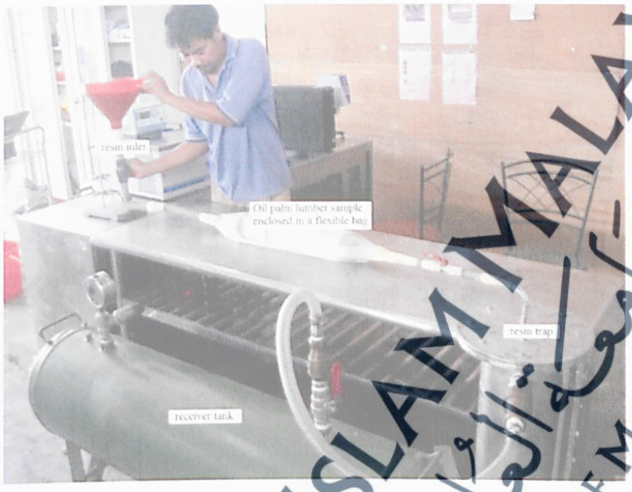


Figure 3.6: A prototype vacuum infusion system, specially developed for gum rosin impregnation of oil palm lumber.

The vacuum was drawn first prior to the introduction of gum rosin. This operation was employed in order to extract the air from the cavity. Thereafter, a pressure difference was applied between the inlet of the flexible membrane, connected to a resin container under atmospheric pressure, and the outlet of the flexible membrane was connected to a pump under vacuum. Once a complete vacuum was achieved, the resin was literally sucked through the OPL by a reduction in vacuum pressure at the outlets, and while keeping the pressure atmospheric (101.3 kPa) at the resin inlets. To circumvent the formation of resin pooling, the flow of gum rosin within the lumber matrix was guided by the oscillating vacuum sequence, is shown in Figure 3.7.

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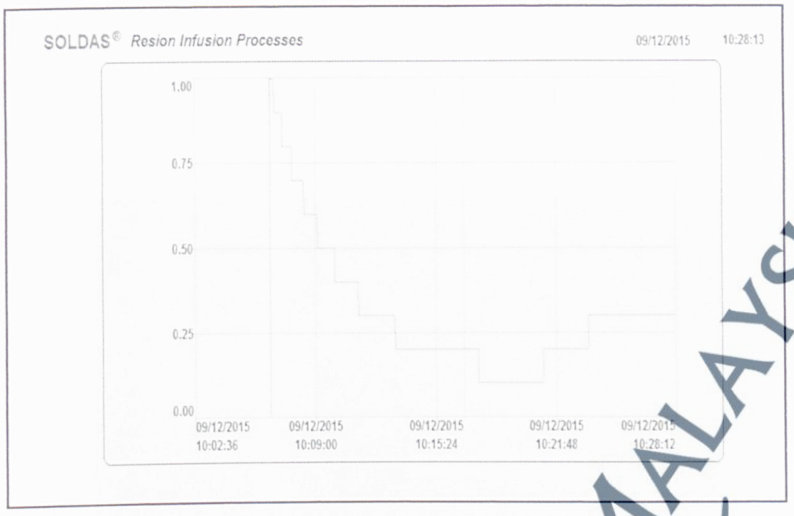


Figure 3.7: Developments of pressure gradient inside a flexible vacuum bag for gum rosin impregnation of oil palm lumber, using the vacuum infusion system

During the infusion, the impregnated part of the OPL sample was subjected to a non-uniform pressure distribution with atmospheric pressure at the inlet and vacuum at the flow front. It was noted that the compaction and the permeability of the wet area tended to vary with position and flow front progression. From observation, the OPL samples of dimensions 100 mm (thick) by 100 mm (wide) by 100 mm (long) was fully wetted with the gum rosin within 2 hours of continuous operations. At the end of the gum rosin filling time, the pressure in the bag was evened out by retaining the vacuum level at the outlets when the gum rosin supply inlet was closed.

The gum rosin-treated sample was then compressed to about 75% of its initial thickness using a cold press (Figure 3.8). This technique will remove excess gum rosin from the sample and to make it more compacted. To complete cross-

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linking of the gum rosin (Kamarudin *et al.*, 2007), the densified gum rosin-treated OPL sample was cured under pressure in an electric oven at 60 ± 5°C for five days.


Figure 3.8: A densification process showing the compression of gum rosin -treated oil palm lumber under static load

### 3.3.3.3 Determination of the gum rosin penetration

Small cubic sample (in the form of a square cross-section of side 5 mm and length along the vascular bundles of 10 mm) was cut across the longitudinal direction with a razor-sharpen knife. The sample was mounted on the stub using double-sided copper tape followed by sputter coating with a 38 nm layer of gold in a vacuum evaporator in order to give it electrical conductivity for scanning electron microscopy (SEM) observation.

In the SEM, the stub (with the specimen) was tilted and rotated in all directions in order to increase the area scanned by the beam, and therefore, creates perspective in the image. The penetration of gum rosin in the lumber matrix was

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determined from the respective photomicrographs. A Hitachi S2700 SEM operated at 15kV was used for the study.

### 3.3.3.4 Evaluation of dimensional stability

Dimensional stabilization was quantified by comparing the volumetric swelling coefficients of both the densified gum rosin-treated and untreated (control) samples of OPL. Measurements of antiswelling efficiency (ASE) were carried out by subjected to an exhaustive water-soaked treatment (Rowell and Ellis, 1978). Ten samples were used for dimensional stability test. These samples were dried at 103 ± 2°C for 24 hours, cooled in a desiccator for 3 hours, and the three structural directions measured. After weighing, the specimen was then vacuum-infused with distilled water until it was fully submerged. Vacuum was then released and the specimen was taken out and soaked under water at 20 °C for 24 hours. The water-soaked dimensions were weighed and measured at the same point as the initial measurements.

### 3.3.3.5 Antiswelling efficiency

An antiswelling efficiency (ASE) value of 0 percent indicates that the treatment imparted no dimensional stability, while a value of 100 percent means that shrinkage and swelling were completely eliminated. The ASE imparted by the treatment was computed by using the equation (Mohd Saiful *et al.*, 2012):

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$$ASE\ (\%) = \frac{S_c - S_t}{S_c} \times 100 \quad \text{(Equation 3.3)}$$

where $S_c$ is the volumetric swelling coefficient of the match-sample devoid of gum rosin, $S_t$ is the volumetric swelling coefficient of densified gum rosin-treated sample of OPL.

The volumetric swelling coefficient ($S_{VC}$) was calculated by using the following equation:

$$S_{VC}\ (\%) = \frac{V_w - V_d}{V_d} \times 100 \quad \text{(Equation 3.4)}$$

where $V_w$ is the volume of OPL sample after soaking in $m^3$ while $V_d$ denotes the volume of respective oven-dry of OPL sample in $m^3$ prior to soaking.

### 3.3.3.6 Reduction in water absorption

The reduction in water absorption ($W_R$) was computed by using the equation (Mohd Saiful *et al.*, 2012):

$$W_R\ (\%) = [(M_c - M_t) / M_c] * 100 \quad \text{(Equation 3.5)}$$

where $M_c$ is water absorption of the matched OPL sample devoid of gum rosin while $M_t$ is water absorbed of the densified gum rosin-treated OPL sample.

The water absorption ($W_A$) was measured by using the equation as follows:

$$W_A\ (\%) = [(W_g - W_d) / W_d] * 100 \quad \text{(Equation 3.6)}$$

where $W_g$ is the weight of the OPL sample in g after soaking, while $W_d$ is the oven-dry weight of OPL sample in g, prior to soaking.

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### 3.3.4 Bending strength test

Strength and stiffness in bending are generally expressed as modulus of rupture (MOR) and modulus of elasticity (MOE), respectively. These properties were determined by three-point bending in accordance with ISO 3133-1975 (1975) procedures. Testing was performed in the ambient condition at 20 ± 3 °C and relative humidity of 65 ± 3% using a Zwick Testing Machine, model NNM 356. The orientation of vascular bundles in test samples was perpendicular to the direction of loading. The loading head was moved at a speed of 5 mm $min^{-1}$ on the center of a test span of 280 mm.

The MOR and MOE were calculated using the equations as follows (ISO 3133-1975):

$$\text{MOR (N mm}^{-2}) = \frac{3P_{max} \times L}{2b \times h^2} \quad \text{(Equation 3.7)}$$

$$\text{MOE (N mm}^{-2}) = \frac{P \times L^3}{4\Delta \times b \times h^3} \quad \text{(Equation 3.8)}$$

where b is the breadth of the OPL in mm, h is the height of the OPL sample in mm, L is the supporting span of OPL sample in mm, $P_{max}$ is the maximum load when the beam is broken in N, P is the load within the proportional deflection in N, and Δ is the deflection at mid-length below the proportional deflection limit in mm.

## 3.4 Statistical Analysis

In general, data summaries were given in tabular form. The values stated represent the mean ± the standard error of the test results obtained. Calculations were performed with a computer spreadsheet software (Microsoft Office Excel 2010 and SPSS 16.0 for Windows®). Statistical comparisons between the groups were

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conducted using an analysis of variance (ANOVA) and comparison among means using the Scheffe's method.

### 3.4.1 Analysis of variance

Analysis of variance (ANOVA) was used to test for differences among sample means and differences among linear combination of means. A simple application of the ANOVA was to test whether two or more sample means could have been obtained from populations with the same parametric mean. The purpose of the ANOVA was to estimate the true differences among the group means. Any single variate can be decomposed as follows (Sokal and Rohlf, 1995):

$$Y_{ij} = \mu + \alpha_i + \varepsilon_{ij} \quad \text{(Equation 3.9)}$$

where $i = 1, \ldots a$, $j = 1, \ldots n$, $\varepsilon_{ij}$ represents an independent, normally distributed variable with mean $\varepsilon_{ij} = 0$, and variance $\sigma^2_\varepsilon = \sigma^2$. Therefore a given reading is composed of the grand mean, $\mu$ of the population, a fixed deviation $\alpha_i$ of the mean of group $i$ from the grand mean $\mu$ and a random deviation $\varepsilon_{ij}$ of the $j$th individual of group $i$ from its expectation, which is $(\mu+\alpha_i)$.

### 3.4.2 Comparison among means

This post-hoc statistical test was used to determine a pair of means that are not from the same population are different from each other and whether the means can be divided into groups that are significantly different from each other. The Scheffe's method for comparison among mean are as follows (Sokal and Rohlf, 1995):

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$$\left| Y_2 - Y_1 \right| \pm [(t-1)*F(\alpha; t-1,v)*(2s^2/n)]^{1/2} \quad \text{(Equation 3.10)}$$

where $Y_1$ and $Y_2$ are the sample means; $s^2$ is the error mean square with $v$ degrees of freedom: F ($\alpha$; $t$ -1,$v$) is the upper (100$\alpha$) % point of F-distribution with ($t$-1) and $v$ degrees of freedom.

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