

CHAPTER 3

EXPERIMENTAL PROCEDURES

3.1 Materials

3.1.1 Coconut wood

The small sheets of coconut wood were obtained from coconut estate and sawn into specimens of 10 x 10 x 30 mm (tangential x radial x longitudinal directions) for compression tests ; 20 x 20 x 10 mm for termite tests ; 25 x 5 x 100 mm for flexural strength tests ; 25 x 5 x 25 mm for dimensional stability tests.

3.1.2 Monomers

Commercial grade styrene and methyl methacrylate were obtained from Siam Chemical Industry Co., Ltd. and used without further purification.

3.1.3 Initiators

Methyl ethyl ketone peroxide (MEKP) was obtained from Siam Chemical Industry Co., Ltd. with tradename, Butanox M-60.

3.1.4 Cross-linker

Divinylbenzene from Fluka is used as cross-linker in the experiment.

ศูนย์วิจัยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

3.2 Apparatus and equipment

1. Vacuum Chamber : 8 inches diameter vacuum dessicator
2. Vacuum Pump : SM – 1/2 R, 1430 RPM.
3. Electric saw : Pehaka, England
4. Venier : Mitutoyo, Japan
5. Sandpaper : Water proof No.80
6. Vacuum oven : Mutter, Germany
7. Balance : Mettler PJ3600, Switzerland
8. Universal Testing Machine : Houndfield 1000 N, England
9. Scanning Electron Microscope : JSM – 6400, JEOL Co., Ltd., Japan
10. Dessicator

3.3 Experiment Procedures

3.3.1 Preparation of coconut wood polymer composites

3.3.1.1 Preparation of wood specimens

Wood specimens were prepared to the test specimens size using electric saw. The rough surface was polished by sandpaper in order to remove the woolly fiber and made smooth surface.

3.3.1.2 Preparation of impregnation solution mixture

Styrene (ST) and/or methyl methacrylate (MMA) was weighed in a vessel. Then the desired amount of initiator was added and mixed homogenously.

3.3.1.3 Impregnation Process

Five wood specimens were first oven dried to constant weight at 105 °C and placed in a chamber that was evacuated (500 mmHg) at 30 °C for a specific time. The monomer mixture containing 2% by wt. of initiator was introduced into the chamber until the wood sample were covered. After the specific time of impregnation, vacuum was released and the impregnation process carried out

for another 30 min. Finally, wood samples were removed from the chamber and wiped to remove excess monomer from wood surface, then weighed immediately to determine monomer intake. The wood samples were wrapped in aluminium foil and thermally polymerized at 70 °C for 24 hours. After unwrapping, the samples were again oven dried at 105 °C overnight to remove unreacted monomer. The specimens were weighed to determine the polymer loading.

3.3.2 The study of the factors influencing in the preparation of coconut wood polymer composites

3.3.2.1 Effect of evacuating time on properties

Coconut wood polymer composites were prepared by impregnation of solution mixture that consist of 99% styrene and 1% by wt. of MEKP in monomer solution. The evacuating time was varied as 0.25, 0.5, 1 hour for low density wood, 0.5, 1, 2 hours for medium density wood and 1, 2, 3 hours for high density wood. The impregnation parameter were as follows: 0.5 hour soaking time, 500 mmHg evacuating pressure and 70 °C curing temperature.

3.3.2.2 Effect of soaking time on properties

Coconut wood polymer composites were prepared by impregnation of solution mixture consisted of 99% styrene and 1% by wt. of MEKP in monomer solution. The impregnation parameters were as follows: 0.5, 2, 2 hours evacuating time for low, medium and high density wood respectively, 500 mmHg evacuating pressure and 70 °C curing temperature. The soaking time was varied as 0.5, 1, 4 hours for all density.

3.3.2.3 Effect of MEKP content on properties

Coconut wood polymer composites were prepared by impregnation of a solution mixture consisted of styrene and MEKP. Different initiator contents as 1, 2, 3, 4 % by wt. in monomer solution were studied and the impregnation parameters were as follows: 0.5, 2, 2 hours evacuating time for low, medium and high density wood respectively, 1, 4, 4 hours soaking time for low, medium and high density wood respectively, 500 mmHg evacuating pressure and 70 °C curing temperature.

3.3.2.4 Effect of curing temperature on properties

Coconut wood polymer composites were prepared by impregnation of a solution mixture consisted of 98% styrene and 2% by wt. of MEKP in monomer solution. The curing temperature was varied as 70 °C and 90 °C temperature and the others impregnation parameters were the same as those mentioned in 3.3.2.3.

3.3.2.5 Effecting of ratios of monomer on properties

Coconut wood polymer composites were prepared by impregnation of a solution mixture consisted of styrene (ST) and/or methyl methacrylate (MMA) and 2% by wt. of MEKP in monomer solution. The ratios of monomers were varied as 100/0%, 70/30%, 50/50%, 30/70% and 0/100% of ST/MMA. The others impregnation parameters were the same as those described in 3.3.2.3.

3.3.2.6 Effect of cross-linker content on properties

Coconut wood polymer composites were prepared by impregnation of a solution mixture consisted of 50/50% of ST/MMA, 2% by wt. of MEKP in a monomer solution and cross-linker. Different cross-linker content as 1% and 4% by weight in monomer solution were studied. The others impregnation parameters were the same as those mentioned in 3.3.2.3.

3.3.3 Testing for physical properties

3.3.3.1 Dimensional stability

3.3.3.1.1 Polymer Loading (PL) [1,17]

The wood specimens were oven dried to constant weight at 105 °C and weighed before impregnation. After impregnation, the wood composites specimens were obtained. They were weighed again, then the polymer loading were calculated as follows :

$$\text{Polymer loading (\%)} = \left[\frac{w_1 - w_0}{w_0} \right] \times 100$$

where w_1 = the oven-dry weight of treated wood or wood polymer composites

w_0 = the oven-dry weight of untreated wood

3.3.3.1.2 Density (D)

The weight and dimension or volume of wood composites specimens were determined. Density was calculated using the following formular:

$$\text{Density} = \frac{W}{V} \text{ (g/cm}^3\text{)}$$

Where W = weight of specimens

V = volume of specimens

3.3.3.1.3 Water absorption (WA) or water uptake [1,17]

Wood composites specimens, both control and treated, were immersed in water at 30 °C for various periods. After each soaking periods, the samples were wiped of excess water and weighed. The water uptake was determined for 2, 4, 8, 24, 48, 72 and 144 hours on the basis of oven dry – measurements.

$$\text{Water absorption (\%)} = \left[\frac{w_1 - w_0}{w_0} \right] \times 100$$

where w_1 = weight of the specimens after soaking

w_0 = weight of the specimens before soaking

$$\text{Water-Repellent Effectiveness (\%)} = \left[\frac{D_c - D_t}{D_c} \right] \times 100$$

where D_c = water uptake of control samples immersed in water for the same periods as water uptake.

D_t = water uptake of impregnated wood samples immersed in water for the same periods as control.

3.3.3.1.4 Antiswelling efficiency (ASE) [1,17]

The dimension of wood composites specimens were measured in radial, tangential and longitudinal direction. Thus the volume before soaking in water was obtained and used to calculate the volumetric swelling coefficient (S). After soaking, the specimens were measured again. The volumetric swelling coefficient and antiswelling efficiency were calculated as follows:

$$\text{Volumetric swelling coefficient, (S)} = \left[\frac{V - V_0}{V_0} \right]$$

where V = wood volume after soaking test for 7 days

V_0 = volume of oven-dried specimens before soaking, test for 7 days

$$\text{Antiswelling efficiency (\%)} = \left[\frac{S - S_0}{S_0} \right] \times 100$$

where S = volumetric swelling coefficient for treated wood

S_0 = volumetric swelling coefficient for untreated wood (control)

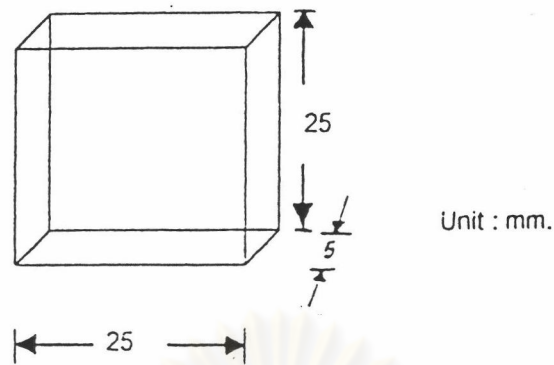


Fig.3.1 Dimension of dimensional stability testing specimen.

3.3.4 Testing for biological property

3.3.4.1 Evaluation of WPC specimens for resistance to termites [ASTM D3345-74]

Wood composite specimens, both control and treated, were weighed before testing and then placed in the bottom of clean containers. 200 g of clean sand was added to each container, distill water was added until saturation point was reached and left them overnight .

$$\% \text{ saturation} = \frac{\text{weight of water}}{\text{oven dry weight of sand}} \times 100$$

$$\% \text{ water to add} = \% \text{ saturation} - 7$$

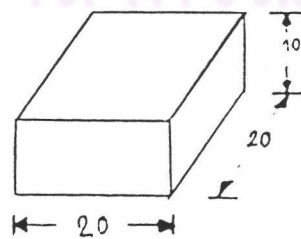


Fig. 3.2 Dimension of termite testing specimen.

The termites were weighed at 1 ± 0.05 g and added into prepared containers that closed tops loosely . The containers were maintained at 25.5 to 27.7 °c for 4 weeks. At the end of the first and fourth weeks, examine the containers and record the presence of tunneling , termite mortality and position of the termites in the container and add distill water to all containers if the moisture content of the sand drops. After 4 weeks disassemble the container and remove and clean the specimens. Examine and visually rate each specimens using the following rating system:

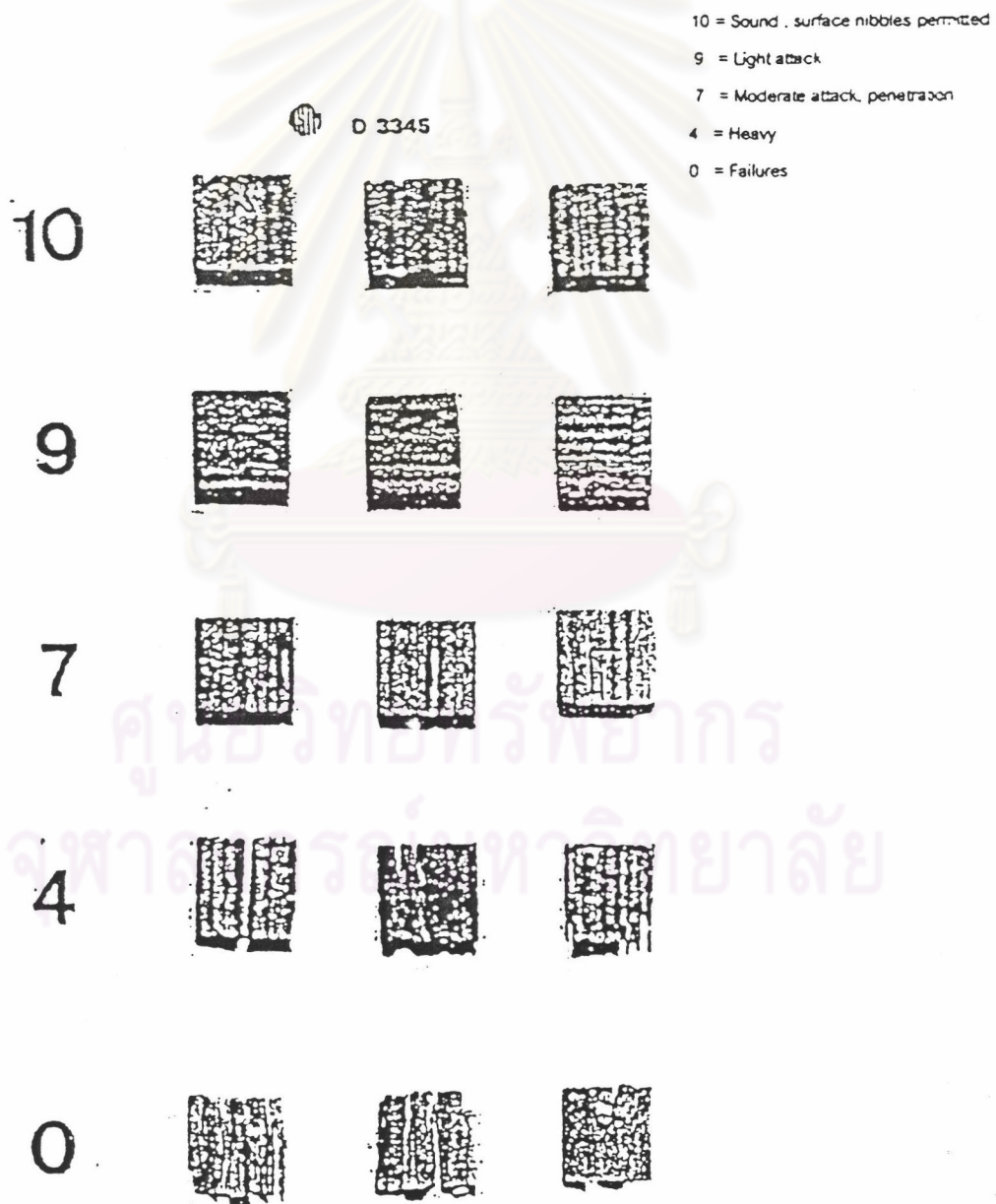


Fig 3.3 Typical Rating of Termite Attack on Test Blocks.

3.3.5 Testing for mechanical properties

3.3.5.1 Flexure stress and Modulus of elasticity (MOE) [ASTM D3043-87]

Width and thickness of wood composites specimens were measured and entered values to the soft-wares of testing machine before running the test. Then flexure stress and modulus of elasticity values were obtained. The MOE corresponds to the slope of the linear portion of the stress-strain relationship from zero to the proportional limit, can be calculated from the stress-strain curve as the change in stress causing a corresponding change in strain, as follows:

$$\text{Modulus of elasticity (MOE)} = \frac{L^3 \Delta W}{4bd^3 \Delta S}$$

Where L= the span between the centers of supports(m)

ΔW = the increment in load (N)

b = the mean width (tangential direction) of the sample(m)

d = the mean thickness (radial direction) of the sample(m)

ΔS = the increment of deflection(m)

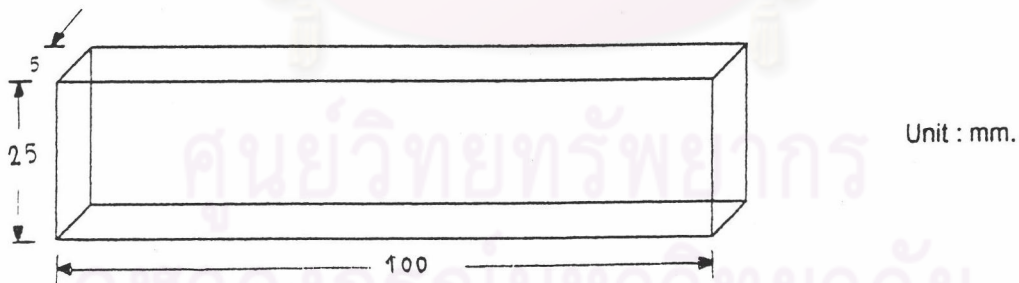


Fig.3.4 Dimension of flexure stress and MOE testing specimen.

3.3.5.2 Compression parallel to grain [ASTM D3501]

The width and thickness of wood composites specimens were measured. Maximum load were obtained after tested. The compression parallel to grain value was calculated as follows:

$$\text{Compression parallel to grain} = \frac{P_{\max}}{a \times b}$$

Where P_{\max} = The maximum load, (N)

a,b = The cross sectional dimensions of the test piece, (mm)

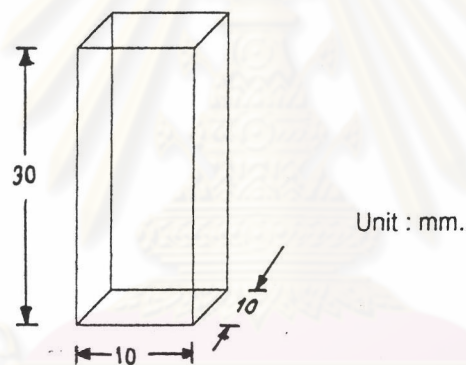


Fig. 3.5 Dimension of compression parallel to grain testing specimen.

3.3.6 Microstructure of WPC specimens

Microstructure of wood-polymer composites specimens were observed by scanning electron microscope and compared with microstructure figure of natural coconut wood. The specimens were dried then coated with gold before scanning observation.