CHAPTER III

EXPERIMENTAL PROCEDURES

3.1 Materials

3.1.1 Rubber wood

The rubber wood was obtained from Bangkok Shuttle Industry Co., Ltd. The samples used in this study were sawn into specimens of $10 \times 10 \times 30$ mm. for compression tests; $20 \times 20 \times 10$ mm. for termite tests; $25 \times 5 \times 100$ mm. for flexural strength tests; $25 \times 5 \times 25$ mm. for dimensional stability tests.

3.1.2 Monomers

3.1.2.1 2-Hydroxy-propylacrylate, commercial grade was obtained from Siam Chemical Industry Co., Ltd.

3.1.2.2 Butylacrylate, commercial grade was obtained from Siam Chemical Industry Co., Ltd.

3.1.2.3 2-Ethyl-hexylacrylate, commercial grade was obtained from Siam Chemical Industry Co., Ltd.

3.1.3 Initiator

Benzoyl peroxide was obtained from Siam Chemical Industry Co., Ltd.

3.2 Apparatus and equipments

- 1. Vacuum Chamber : modified from 8 inches diameter dessicator
- 2. Vacuum Pump : SUPERPOWER, Thailand
- 3. Vernier : MITUTOYO, Japan
- 4. Vacuum Oven : MUTTER, Germany

- 5. Electric Saw : PEHAKA, England
- 6. Universal Testing Machine : HOUNDFIELD H10KM, England
- 7. Balance : METTLER, Switzerland
- 8. Scanning Electron microscopy : JSM-6400, JEOL Co., Ltd., Japan
- 9. Sandpaper : WATER PROOF NO.80
- 10. Dessicator

Apparatus for vacuum impregnation is shown in Figure 3.1



Figure 3.1 Apparatus for vacuum impregnation

- A Vacuum dessicator
- B Plastic or glass treatment beaker
- C Test wood blocks
- D Glass or suitable weight
- E Treating solution
- F Polyethylene tubing
- G Three-way stopcock
- H Flask containing treating solution
- I Line to vacuum source

3.3 Experimental Procedures

3.3.1 Preparation of rubber wood-polymer composites

3.3.1.1 Preparation of wood specimen

The wood specimens used for testing were prepared from clear, defectfree rubber wood. Test specimens were sawed in the dimension in 3.1.1 and oven dried at $105^{\circ}C$ for 24 hours prior to treatment.

3.3.1.2 Determination of moisture content and density of specimens

Each specimen was weighed and then reweighed after drying over night in oven at 105° C. Moisture content was calculated as the percentage of moisture based on the weight of wood.

Moisture Content (%) =
$$\frac{W_0 - W_1 \times 100}{W_1}$$

where W_0 = weight before drying W_1 = weight after drying

The volume of each sample was determined. Density was calculated using equation below.

$$Density = W_1 / V (g/cm')$$

where

V = volume of specimen after drying

3.3.1.3 Preparation of impregnation monomer solution

The monomer solutions were prepared with 2-hydroxy-propylacrylate (2-HPA), butylacrylate(BA) and 2-ethyl-hexylacrylate(2-EHA) in beakers. Then, the desired amount of benzoyl peroxide was added and mixed homogeneously.

3.3.1.4 Preparation of rubber wood-polymer composites

All wood specimens for impregnation or testing were first oven dried at 105[°]C for 24 hours and placed in a vacuum chamber to evacuate at room temperature for desired time. The monomer and benzoyl peroxide as a catalyst were introduced into the vacuum chamber until the wood samples were covered. After the desired soaking time was reached, the vacuum was releases. Finally, wood samples were removed and wiped out to remove excess monomer mixture from wood surface (polymerization was usually begun during the impregnation process). The wood samples were wrapped in aluminium foil and thermally polymerized at 90[°]C for 24 hours. The apparatus for vacuum impregnation is shown in Figure 3.1.

3.3.2 The study of the factors influencing in the preparation of rubber woodpolymer composites

3.3.2.1 Effect of evacuating time on properties

Rubber wood-polymer composites were prepared from prepolymer solutions containing materials as follows: 2-HPA with 1 phr initiator, BA with 1 phr initiator and 2-EHA with 1 phr initiator. The impregnation parameters were as follows: 4 hours soaking time and various evacuating times at 0.5, 1, 2, 3 hours.

3.3.2.2 Effect of soaking time on properties

Rubber wood-polymer composites were prepared from prepolymer solutions containing materials as follows: 2-HPA with 1 phr initiator, BA with 1 phr initiator and 2-EHA with 1 phr initiator. The impregnation Parameters were as follows: 2 hours evacuating time and various soaking times at 1, 2, 3, 4, 6 hours.

3.3.2.3 Effect of initiator contents on properties

Benzoyl peroxide content as 0.5, 1, 2 and 3 phr was added to prepolymer solution. The impregnation parameters were as follows: evacuating time was 2 hours and soaking time was 4 hours.

3.3.3 Testing for physical properties

3.3.3.1 Polymer loading

Before impregnation, the specimens were dried in an oven at 105^oC overnight and weighed. After impregnation, the wood composite specimens were obtained. They were weighed again and the polymer loading were calculated as follows:

Polymer loading (%) = $\frac{W_t - W_0}{W_0} \times 100$

where

 W_t = weight of treated wood W_0 = weight of untreated wood (oven dry)

3.3.3.2 Density of specimens

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

Density = $W / V (g/cm^3)$

where

W = weight of specimensV = volume of specimens

3.3.3.3 Water Absorption (WA)

Wood composite specimens were immersed in water at room temperature for 7 days. After that period, the specimens were wiped of excess water and weighed. The water absorption was determined on the basis of oven dry measurements.

Water absorption (%) =
$$\frac{W_1 - W_0}{W_0} \times 100$$

where W_1 = weight of specimens after water soaking

 W_0 = weight of specimens before water soaking

3.3.3.4 Antiswell efficiency (ASE)

The dimensions of wood composite specimens were measured in radial, tangential and longitudinal directions. Thus, the volume before soaking was measured and used to calculate the volumetric swelling coefficient (S). After soaking, the specimens were measured again. The volumetric swelling coefficient and antiswell efficiency were calculated as follows:

Volumetric swelling coefficient (S) = $\frac{V_1 - V_0}{V_2}$

where

 V_1 = wood volume after water soaking V_0 = wood volume before water soaking

Antiswell efficiency (ASE) = $\frac{S_1 - S_0}{S_0} \times 100$

where

 S_1 = volumetric swelling coefficient of untreated wood S_0 = volumetric swelling coefficient of treated wood

3.3.4 Mechanical properties

Mechanical properties were measured as follows:

3.3.4.1 Flexure stress and modulus of elasticity (MOE) [ASTM D3043-87]

The width and thickness of wood composite specimens were measured and used as input data to the software of testing machine before running the test. Then, flexure stress and modulus of elasticity values were determined. The MOE, corresponding to the slope of the linear portional limit, can be calculated from the stress-strain curve as the change in stress causing a corresponding change in strain, as follows:

Modulus of elasticity (MOE) = $L^3 \Delta W$ 4bd³ ΔS

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 in deflection (m)

The dimension of testing specimen is shown in Figure 3.2 and the testing machine is shown in Figure 3.3.



Figure 3.2 Dimension of specimen for flexure stress and MOE testing



Figure 3.3 The testing machine of flexure stress and MOE

3.3.4.2 Compression parallel to grain [ASTM D3501]

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

Compression parallel to grain = P_{max} a x b

where P_{max} = the maximum load (N)

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

The dimension of testing specimen and tested samples are shown in Figures 3.4 and 3.5.



Figure 3.4 Dimension of a specimen for compression parallel to grain testing



Figure 3.5 Tested samples from compression parallel to grain testing

3.3.5 Termite Resistance [ASTM D3345]

In this study, three types of wood, Makah-mong, Red wood, and natural rubber wood were compared with treated rubber wood specimens. Prior to test, the container was prepared by washing and rinsing with antiseptic solution and dried. Each specimen was prepared as 20 (T) x 20 (R) x 10 (L) mm. and then weighed before testing. The prepared specimens were placed in the bottom of container. The cleaned sand (200 g.) was added in the container, followed by sufficient distilled water as determined by the equation below:

% water to add = % saturation -7.0

calculate the percent saturation as follows:

% saturation = (weight of water / oven dry weight of sand) x 100

After addition of water, the container was left overnight. The termites was weighed to 1 \pm 0.05 g. and added into prepared container with loosely closed tops. The container was maintained at room temperature for 4 weeks (Figure 3.7). After 4 weeks, the container was disassembled and the wood blocks were removed and cleaned. The test blocks were weighed again for %weight loss and examined visually at each block using the following rating system in Figure 3.6.

- 10 = Sound, surface nibbles permitted
- 9 = Light attack
- 7 = Moderate attack, penetration
- 4 = Heavy
- 0 = Failure



Figure 3.6 Typical ratings of termite attack on test blocks



Figure 3.7 Apparatus of termite testing

3.3.6 Microstructure of WPC Specimens

Microstructure of rubber wood-polymer composite specimens were observed by scanning electron microscope and compared with microstructure figure of natural rubber wood. The specimens were dried, then coated with gold before scanning for the observation.