

## CHAPTER III

### EXPERIMENT

#### 3.1 Materials

Kenaf fiber (*Hibicus sabdariffa* Linn. *Var. altissima*) was supplied by N.E.P. Real Estate Property and Industry Co., Ltd., Thailand. The fibers were in the form of combed fiber bundles.

Phenol (>99.5%), p-formaldehyde (>95%), aniline (99.5%), and bisphenol-A (>97%) were purchased from Merck Company. Unsaturated polyester (UPE) resin, accelerator, and catalyst were supplied by Siam Chemical Industry Co., Ltd. All chemicals were used without further purification.

#### 3.2 Instruments

##### 3.2.1 Fourier Transform Infrared Spectrometer (FTIR)

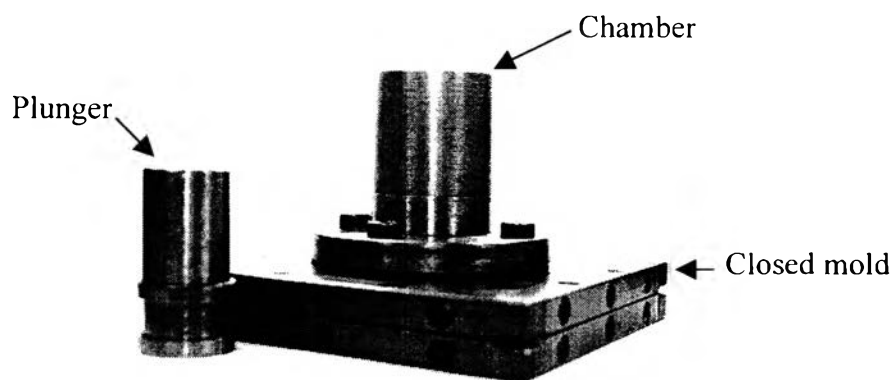
FTIR spectra were obtained on a Bruker Equinox55 Spectrophotometer with 32 scans at a resolution of 4 cm<sup>-1</sup> and a frequency range of 4000-400 cm<sup>-1</sup>.

##### 3.2.2 Nuclear Magnetic Resonance Spectrometer (NMR)

<sup>1</sup>H-NMR spectra were collected on a Bruker 400 MHz Spectrometer.

### 3.2.3 Resin Transfer Mold

The stainless steel mold for making the composites is shown in Figure 3.1. It consists of two mold plates with dimensions of 180×180 mm, a chamber which can hold 94.2 ml of resin, and a plunger for pushing down the resin in the chamber. The volume of mold cavity is 150×150×3 mm.



**Figure 3.1** Mold for making composites by the resin transfer molding technique.

### 3.2.4 Instron Universal Testing Machine

Tensile and flexural tests of the composite samples were carried out on an Instron Universal Testing Machine model 4206.

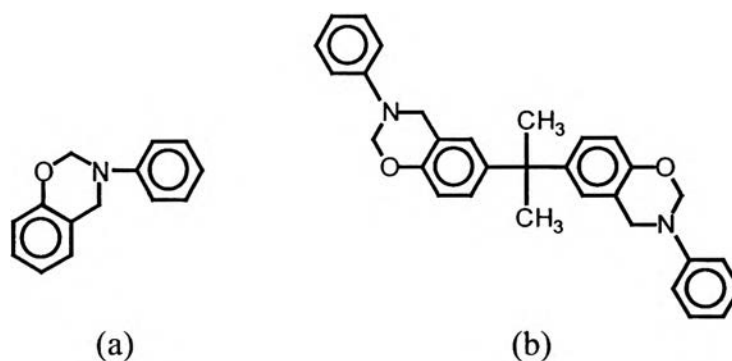
### 3.2.5 Zwick Pendulum Impact Tester

Impact tests of the composite samples were performed on a Zwick Pendulum Impact Tester model 5113.

### 3.3 Methodology

#### 3.3.1 Monomer preparation

Two benzoxazine monomers were synthesized for this study. Their chemical structures are shown in Figure 3.2. The IUPAC notation of these compounds are 3,4-dihydro-3-phenyl-2H-1,3-benzoxazine (abbreviated hereafter as Ph-a) and bis(3,4-dihydro-2H-3-phenyl-1,3-benzoxazinyl) isopropane (abbreviated as BA-a).



**Figure 3.2** Molecular structures of monomers used for the synthesis of polybenzoxazine resins: (a) Ph-a monomer (b) BA-a monomer.

##### 3.3.1.1 *Ph-a monomer*

This monofunctional benzoxazine monomer is based on phenol and aniline. The molar ratio of phenol:p-formaldehyde:aniline was 1:2:1. Phenol, p-formaldehyde and aniline were added to a reaction flask, preheated to 80°C, in the absence of solvent. The mixture was then heated and stirred at 100°C for 30 minutes. The reaction produced Ph-a monomer in the form of a yellow liquid.

### 3.3.1.2 *BA-a monomer*

This bifunctional benzoxazine monomer is based on bisphenol-A and aniline. The molar ratio of bisphenol-A:p-formaldehyde:aniline was 1:4:2. The synthesis of this monomer was similar to that described above for monomer Ph-a. The BA-a monomer was obtained as a pale yellow solid.

Characterization of these two benzoxazine monomers was carried out using Fourier transform infrared spectroscopy (FTIR) and nuclear magnetic resonance spectroscopy (NMR).

## 3.3.2 Preparation of Resins

### 3.3.2.1 *Polybenzoxazine resin*

Ph-a and BA-a monomers were degassed in a vacuum oven at 100°C for 6 hours to remove any remaining volatiles. The two monomers were then mixed at the required ratio by stirring together at 100°C for 5 minutes. The resultant resin mixture was again degassed in a vacuum oven at 100°C for 30 minutes.

### 3.3.2.2 *Unsaturated polyester resin*

UPE resin was first diluted with 10 wt% styrene monomer to obtain a resin mixture of suitable viscosity. Cobalt octoate (accelerator) 0.5 wt%, and methyl ethyl ketone peroxide (catalyst) 1 wt%, were added and mixed with the resin just before the mixture was used.

## 3.3.3 Resin Transfer Molding of Composites

Kenaf fibers were dried in an oven at 105°C for 4 hours to reduce the moisture content and then cut into 15 cm lengths. The cavities of the RTM mold was first coated with silicone. Kenaf fibers, weighing 20 wt%

of the composite, were laid unidirectionally in the mold which was then closed. Benzoxazine resin was poured into the chamber and a plunger used to force the resin into the mold. A hydraulic press was used to press the plunger (Figure 3.1). The composite was then cured at a higher temperature between 150-170°C for a fixed time. The cured composite had a thickness of 3 mm.

#### 3.3.4 Monomer Characterization

The as-synthesized benzoxazine monomers were characterized using FTIR. Neat technique on ZnSe cell and sample powder pressed into a KBr pellet were used. The benzoxazine monomers were also characterized using NMR. Deuterated chloroform was used as a solvent and tetramethylsilane (TMS) was added as a standard.

#### 3.3.5 Mechanical Testing

Tensile and flexural tests of the composite samples were carried out on an Instron universal testing machine according to ASTM D638-96 and D790-96a, respectively. Tensile tests were conducted using five dumbbell shape specimens with 140 mm in length, a crosshead speed of 5 mm min<sup>-1</sup> and a gauge length of 50 mm. Five rectangular test specimens of each sample measuring 80×12.7×3 mm were used at a crosshead speed of 1.28 mm min<sup>-1</sup> in a three-point loading, fitted with a 5 kN load cell. Impact tests were performed on a Zwick pendulum impact tester in accordance with ASTM D256-97. Five rectangular test specimens with dimensions of 63.5×12.7×3 mm were used.

#### 3.3.6 Determination of Water Absorption

Water absorption of the composite specimens was measured according to ASTM D570-95. Five specimens were cut into pieces of 76.2×

25.4×3 mm. They were dried in an oven for 24 hours at 50°C, cooled in a desiccator, and immediately weighed. After that, they were immersed in distilled water for a set time and temperature. The specimens were then periodically removed, wiped dry, weighed, and immediately returned to the water bath. The amount of absorbed water was calculated as the difference between the mass at each of these measurement times and the initial conditioned mass.