

## CHAPTER III

### EXPERIMENTAL SECTION

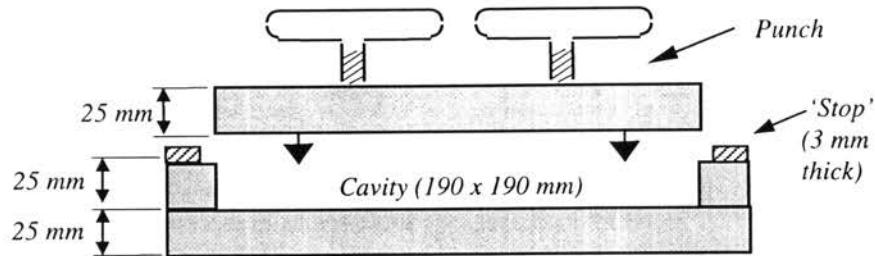
#### 3.1 Materials

Kenaf fiber (*Hibiscus 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. Non-ionic detergent, Tinovetin JU, was supplied by Ciba-Geigy. Vinyltriethoxysilane (VTS) was purchased from Fluka.  $\gamma$ -Methacryloxypropyltriethoxysilane ( $\gamma$ -MPS) was supplied by Thai Vetrotex Co., Ltd. Unsaturated polyester resin and catalysts were supplied by Siam Chemical Industry Co., Ltd. All chemical reagents were used as received.

#### 3.2 Instruments

##### 3.2.1 Compression Molding Machine

The thickness of the composites was controlled by placing the mold, on which 3 mm thick steel stops were placed, in a Wabash compression press model V50h. A force of 5 tons was applied to the mold. The mold for making the composites is shown in Figure 3.1.



**Figure 3.1** Mold for making composites by hand lay-up followed by compression molding.

### 3.2.2 Instron Universal Testing Machine

Mechanical testing of the composite samples was carried out on an Instron Universal Testing Machine model 4206.

### 3.2.3 Scanning Electron Microscope (SEM)

The surface of the fractured specimens from tensile testing was examined using a JOEL scanning electron microscope model 520. Micrographs of the fractured surfaces were made using a voltage of 15-25 kV and 35× magnification.

## 3.3 Methodology

### 3.3.1 Preparation of Fiber

#### 3.3.1.1 *Fiber Cleaning*

The fibers were treated with hot (70°C) non-ionic surfactant solution (3 wt%) at a liquor ratio of 100:1 for 45 min. The samples were then washed with distilled water 3 times. The process was repeated twice to ensure

thorough cleaning of the fibers. Finally, the fibers were dried at 80°C for 24 hours.

#### 3.3.1.2 *Fiber Treatment*

Silanol solutions were prepared from the silanes using distilled water. The pH of the solutions were adjusted to 3.5 with hydrochloric acid. Solutions were kept in PP flasks and left to completely hydrolyze in a shaking water bath at 30°C for 1 hour. Fibers were treated in batches by soaking about 40 g of the fibers in 1200 ml of the silanol solution for 1 hour at 30°C. The solution was then removed by passing through a suction filter and the fibers left to dry at room temperature for 12 hours before being dried to constant weight in a vacuum oven at 70°C. The amount of silanol adsorbed was determined by the difference in the dried weight of fibers before and after the treatment. In this work, the concentrations of the silanol solutions used were 1, 3, and 5 wt% respectively.

#### 3.3.2 Hand Lay-Up of Composites

Unsaturated polyester resin was first diluted with 20 wt% styrene monomer to obtain a resin mixture with suitable viscosity for laminating. Cobalt octoate (accelerator) 0.3 wt%, and methyl ethyl ketone peroxide (catalyst) 1 wt%, were added and mixed with the resin just before the mixture was used. Each composite was prepared using 8 layers of unidirectional oriented fibers. The fiber layers were laid one by one in a mold having cavity dimensions of 190 × 190 × 25 mm (Figure 3.1). After applying each layer of fiber, the prepared resin was added and stippled into the fibers with a brush to remove entrapped air and saturate the kenaf fibers. A roller was then used to consolidate the mat and remove any entrapped air before the next layer was introduced. After the process was complete, the mold punch was placed in position on the laminate and the mold slowly closed and compressed down to

thickness stops in the compression press. The composite was molded at room temperature under a closing force of 5 tons for 1 hour followed by curing in an air-circulating oven at 60°C for 1 hour and 80°C for 3 hours.

### 3.3.3 Mechanical Testing

Tensile tests were conducted using a crosshead speed of 5 mm min<sup>-1</sup> and a gauge length of 50 mm in accordance with test procedure ASTM D 638-96. Flexural tests, using three point bending mode, were performed in accordance with ASTM D 790-96a. Rectangular test specimens measuring 80 × 12.7 × 3 mm were used and tested at a crosshead speed of 5 mm min<sup>-1</sup>.

### 3.3.4 Determination of water adsorption

Water adsorption of the composite specimens was measured by soaking them in distilled water for a set time and temperature and the weight gain determined. The tests were carried out at room temperature, 70°C, and 100°C for different durations.