

CHAPTER IV

EXPERIMENTAL DETAILS

4.1 Materials

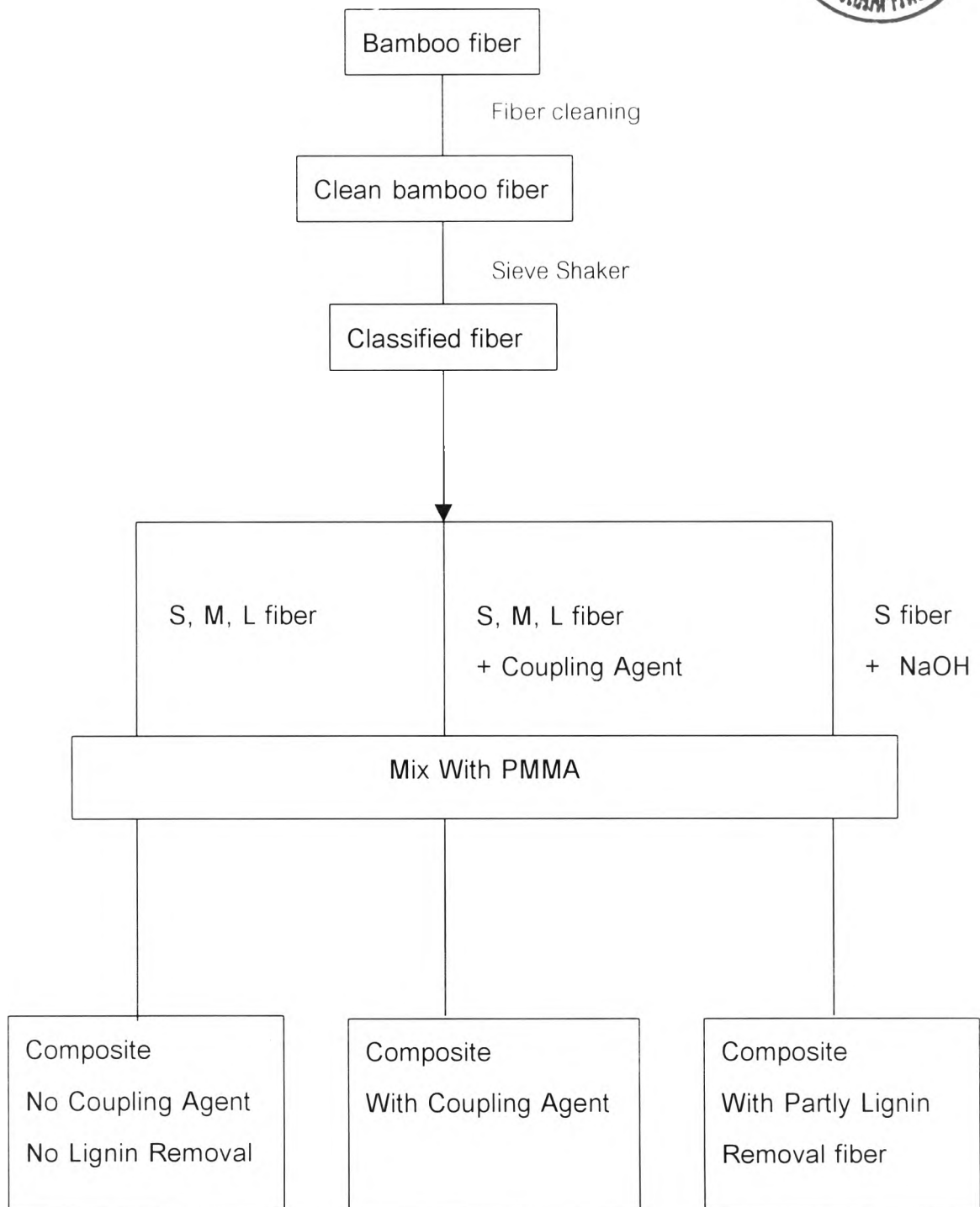
Poly(Methyl Methacrylate (PMMA, trademark: Meliodent Heat Cure Powder) and Methyl Methacrylate Monomer (MMA, trademark: Meliodent Heat Cure Liquid) were used as main components to form matrix in this study. They were both supplied by Heracus Kulzer GmbH & Co. Ltd.

Bamboo Fiber (*Bambusa aspera Schultes*) is a reinforcing element in this research. It was supplied from TAIHEIYO International (Thailand). The fiber was in form of combed fiber bundles. In this study, the cleanness of interfacial surface was important. Thus, non-ionic detergent (Tinovetine JU) was used to ensure that the surface of fiber is clean and there is no affect from dirt. This detergent was supported by Ciba-Geigy.

Normally, the adhesion between the PMMA matrix and bamboo fiber is poor. γ -Methacryloxypropyletrimethoxysilane (γ -MPS) was selected to be used as coupling agent because it is normally used in dental application. It was purchased from Sigma-Aldrich Co., Ltd. Furthermore, the chemical to adjust pH of silanol solution in this study is acetic acid that was purchased from SR Lab Co., Ltd.



4.2 Flow chart of work operation



Note: Size Variation: S / M / L

Fiber Content Variation: 0, 5, 10, 20 wt %

Testing items: Flexural Test / Impact Test / Compressive Test / SEM

4.3 Methodology

4.3.1 Preparation of Bamboo Fiber

4.3.1.1 Fiber Cleaning

The fibers were treated with hot (70°C) non-ionic detergent solution (3-wt %) for 45 minutes with the ratio of 1g of fiber by 100g of solution. The fibers 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.

4.3.1.2 Fiber Size Classification

The fiber was classified into 7 size-groups as the follows:

- a) Size L (1 - 2 mm)
- b) Size M (425-600 micron)
- c) Size S (150-250 micron, normal)
- d) Size S (6-hour treatment of NaOH)
- e) Size S (12-hour treatment of NaOH)
- f) Size S (18-hour treatment of NaOH)
- g) Size S (24-hour treatment of NaOH)

Sieve Shakers (Aperture: 2 mm, 1mm, 600 μm , 425 μm , 250 μm ., 150 μm .) were used to classified the fiber size L, M, and S. In each group, the fiber length was measured by optical microscope (OLYMPUS, model BH-2). The fiber diameter was examined by Scanning Electron Microscope (SEM).

4.3.1.3 Fiber Treatment

Silanol solutions were prepared from the silanes using distilled water. The pH of the solutions was adjusted to 3.5 with acetic acid. Solutions were left to completely hydrolyze in water bath at 50°C for 1 hour. Fibers were treated in batches by soaking about 40 g. of fibers in 1200 ml of the silanol solution for 1 hour at 50°C. The solution was then removed by passing through a suction filter and the fibers left to be dry at room temperature for 12 hours before being dried to constant weight in an 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. The concentration of the silanol solution used in this study was 5 wt %

4.3.1.4 Lignin Removal

In this study, lignin was partly removed by treating 30 g of clean fiber in 3000 ml of hot NaOH solution (1 wt %). The temperature was set at 100°C. The treating time was varied as 6, 12, 18 and 24 hours.

4.3.1.5 Bamboo fiber loading

The effect of % bamboo fiber loading on the mechanical properties was also studied in each sample. The amounts of the fiber loading were varied as 0, 5, 10 and 20 wt %.

4.3.2 preparation Blank PMMA

Blank PMMA was made by mixing PMMA powder to MMA monomer at the ratio of PMMA 35 g by MMA 14 ml, then stirring in the melamine cup until it is dough-like.

After that, the dough was formed in the mould and taken in the hydraulic hot-press machine at 70°C for 2 hours and 100°C for 3 hours. The pressure to mould the sample was 250 kg/cm².

4.3.3 Making composite samples

Composite samples were made by mixing treated or untreated bamboo fiber to MMA monomer, and then mixing with PMMA with the same ratio and conditions as 4.3.2.

4.3.4 Cutting the samples

After sample molding was finished, each sample was cut with hand saw and polished as ASTM standard size with Grinder and Polishing machine (BUEHLER METASERV).

4.3.5 Interface determination

The surface of the fiber with and without coupling agent including that of the broken specimen are determined by JOEL scanning electron microscope model JSM-5410 LV.

4.4 Mechanical Tests

4.4.1 Flexural test

Flexural testing was performed by using Instron universal testing machine (LLOYD 2000R, polymer engineering research laboratory, Faculty of Engineering, Chulalongkorn University). The test conditions follow ASTM D790M. The specimen dimensions are 3 mm in thickness, 25 mm in width and 60 mm in length. The specimens arranged in a three-point bending configuration with L/D ratio = 16 were tested at a constant crosshead speed of 1.2 mm/min and 48 mm for supported span. The flexural testing is shown in Figure 4.1

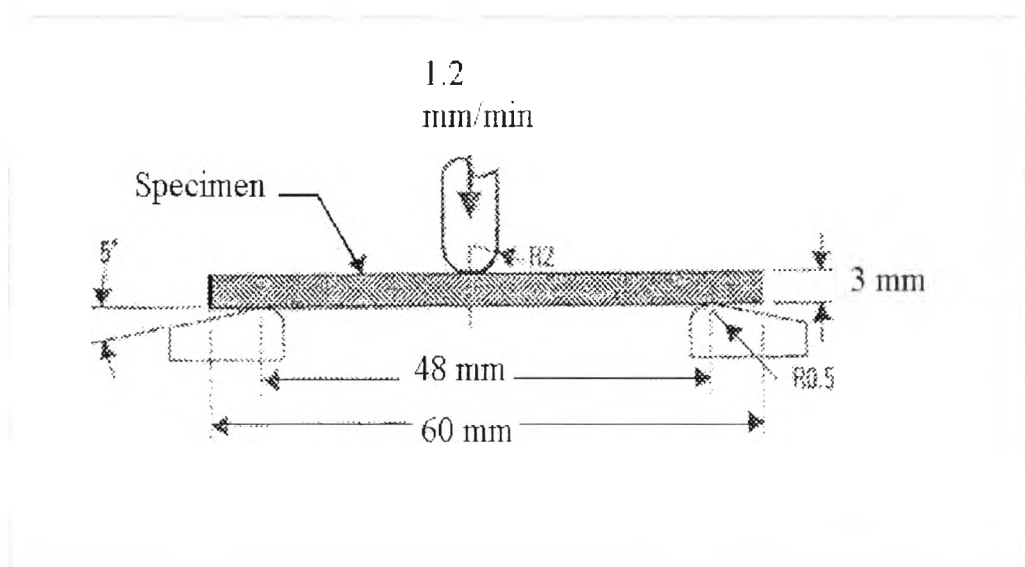


Figure 4.1 Flexural Testing

For flexural test, procedure A of ASTM D790M was selected for PMMA, which undergoes small deflection during test. The results of the tests are the average values of five samples. Raw data are shown in Appendix.

4.4.2 Impact test

Izod impact test was carried out according to ASTM D256 on a (ITR-2000) (National Metal and Materials Technology Center). The dimensions of test specimens are 12.75 mm in width and 63.5 mm in length. Notching machine (RAY-RAN POLYTEST model 1) was used to make a notch at the depth of 2.54 mm. The specimen of Izod type test is shown in Figure 4.2

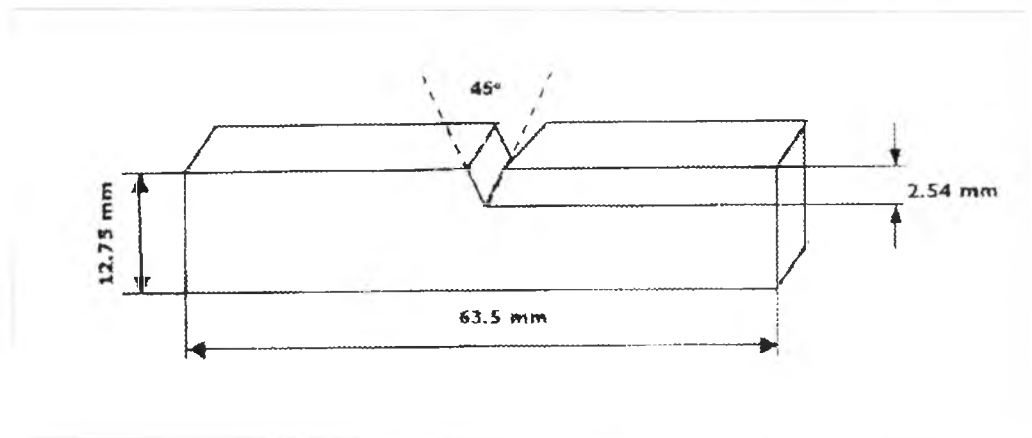


Figure 4.2 Dimensions of Izod Impact test.

The apparatus for Izod impact test is presented in Figure 4.3. The test specimen was clamped into the position that the notched end of the specimen faced the striking edge of the pendulum. The moving hammer was released, allowed to strike the specimen. Impact resistance results are defined as the recorded impact energy (in joules) used to break the test specimen, divided by the specimen area at the notch. The results of the test are the average values of five samples. Raw data of Izod impact test are given in Appendix.

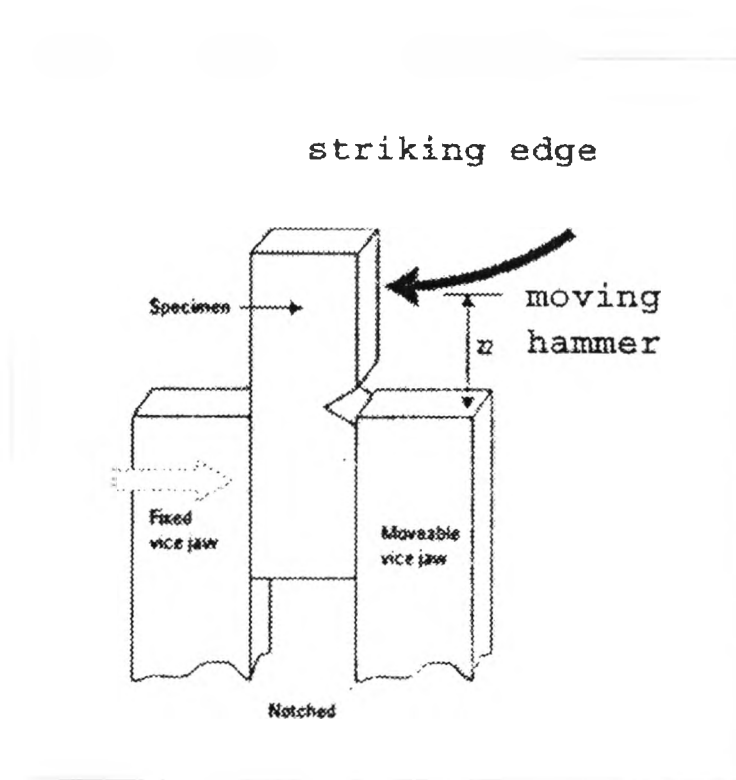


Figure 4.3 The apparatus for Izod impact test.

4.4.3 Compressive Test

Compressive test was carried out according to ASTM D695 on a Instron universal testing machine (LLOYD LR1000K, Department of Material Science, Faculty of Science, Chulalongkorn University). The typical dimensions of test specimens are 12.7 mm x 12.7 mm x 25.4 mm. The specimen is placed between compressive plates parallel to the surface. The specimen is then compressed at a uniform rate. The apparatus of compressive test is shown in Figure 4.4.

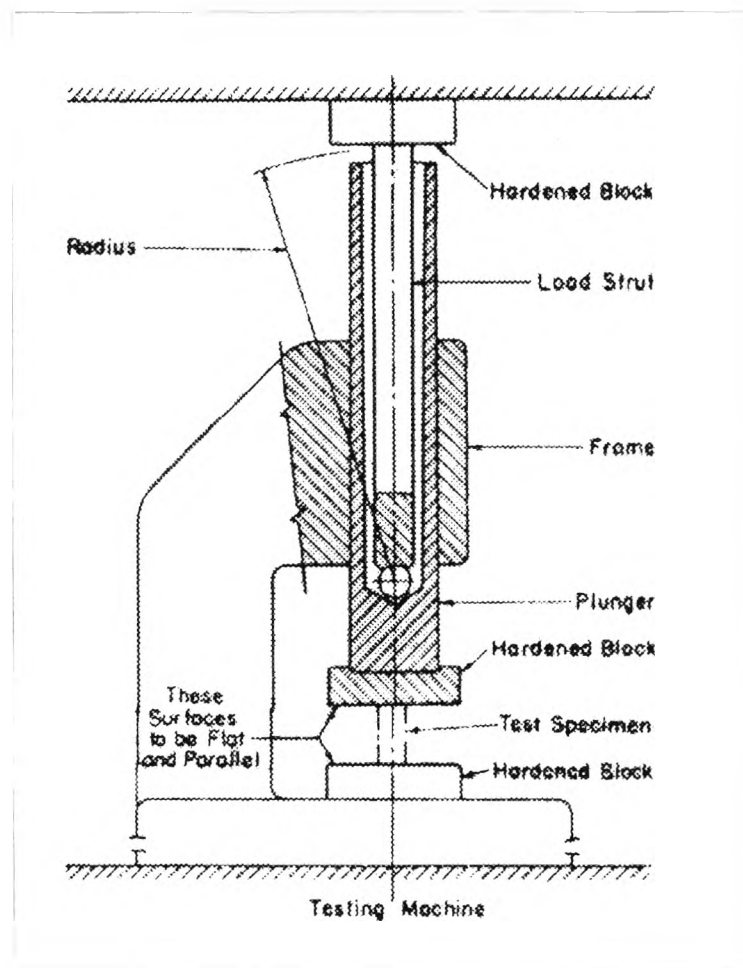


Figure 4.4 Compressive test.

In this study, the results of the tests are the average values of five samples. Raw data are shown in Appendix.