# CHAPTER III
# EXPERIMENTAL

## 3.1 Materials

### 3.1.1 High Density Polyethylene (HDPE)

High density polyethylene of blown film grade (Thai-zex 7000F) was kindly supplied by Bangna Plaschem Co., Ltd. Some physical properties are shown in Table 3.1.

**Table 3.1** Physical property of HDPE.

| **Properties** | **Unit** | **Typical Value** |
|---|---|---|
| Melt Flow Index | g/10min | 0.04 |
| Density | $g/cm^3$ | 0.96 |
| Melt Density | $g/cm^3$ | 0.76 |
| Melting Point | °C | 131 |
| Softening Point | °C | 124 |
| Tensile strength at yield | $Kg/ cm^3$ | 280 |

3.1.2 Fillers

Tapioca starch was kindly supplied by Siam Modified Starch Co.,Ltd.
Rice husk was supported by Thanyakij Nakornprathom Rice Milling.
Burning husk was purchased from Royal Jitralada Project.

## 3.2 Experimental Procedures

3.2.1 Fillers Preparation

*3.2.1.1 Rice Husk Preparation*

Rice husk was washed and dried before it was passed through prehydrolysis with water by autoclaving at 121 °C for 1 hour to remove some water-soluble components such as hemicellulose in rice husk. In order to obtain crude fiber of rice husk, two alkaline extraction were done under high pressure condition at 121 °C for 1 hour to extract alkaline-soluble components such as silica, lignin, and protein. Then the treated rice husk was thoroughly washed with water until the washing solution was neutral to pH paper. The treated rice husk was collected and ground into a size range of 38-53 micron. Finally it was stored in desiccator before use. The flow chart of rice husk preparation is shown in Figure 3.1.

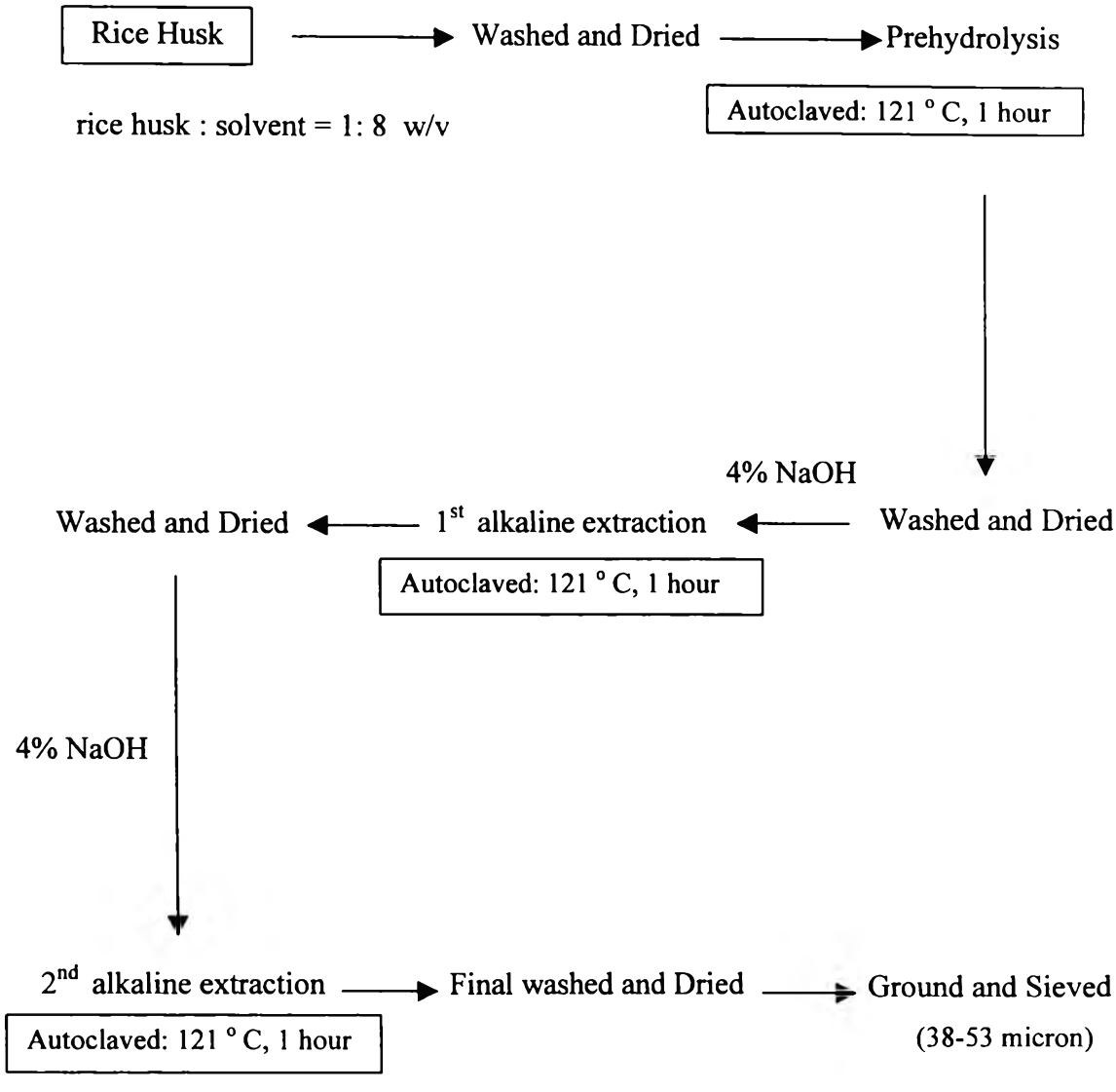


**Figure 3.1** Preparation process of rice husk.

*3.2.1.2 Burning Husk Preparation*

Burning husk was milled and sieved to collect particles size in the size range of 38-53 micron. The burning husk powder was dried in oven at 100 $^{O}$C for 2 hours and then kept in a desiccator before use.

*3.2.1.3 Tapioca Starch Preparation*

Tapioca starch was dried in oven at 100 $^{O}$C for 2 hours to remove the moisture. After that, tapioca starch was stored in a desiccator before use.

### 3.2.2 Filler Density Measurement

The densities of the fillers were measured using a pycnometer. The filler was accurately weighed and put into a pycnometer. Solvent with known density was filled in the pycnometer containing the filler and then reweighed. In this work, ethylene glycol was used as a solvent to measure the density of the fillers. The densities of the fillers were determined from equation 3.1.

$$\rho_f = [\ W_f /(V_{pyc} -(W_{sol} /\rho_{sol}))] \qquad (3.1)$$

Where:

$\rho_f$ is the density of the filler (g/cm$^3$)

$W_f$ is the weight of the filler (g)

$V_{pyc}$ is the volume of the pycnometer (cm$^3$)

$W_{sol}$ is the weight of solvent (g)

$\rho_{sol}$ is the density of solvent (g/cm$^3$)

**Table 3.2** Density of the solvent.

| Solvents | Density Values (g/cm$^3$) |
|---|---|
| Ethylene glycol (20°C) | 1.109 |

### 3.2.3 Thermogravimetric Analysis

In order to determine the decomposition temperature of each filler, thermogravimetric analysis was conducted by heating the fillers from 50 °C to 700 °C at a heating rate of 10 °C/min using a Perkin Elmer thermogravimetric analyzer model TGA 7. The decomposition temperatures of rice husk and tapioca starch were 299.97 °C and 300.80 °C, respectively. The burning husk did not exhibit a decomposition temperature.

### 3.2.4 Polymer Blend Preparation

Mixing of HDPE with the three types of fillers were done using Brabender Plasti-Corder, PL 2100. A rotor speed of 30 rpm and oil bath temperature of 170 $^{O}$ C were used. The batch compositions blend were calculated from equation 3.2.

$$\text{Batch size (g)} = V_c * D_b * F \qquad (3.2)$$

Where:

$V_C$ is the volume of the chamber ($cm^3$)

$D_b$ is the density of the blend ($g/cm^3$)

F is the fill factor of the blending system = 0.8

The batch compositions of the blends are summarized in Table 3.4, 3.5 and 3.6.

**Table 3.3** Batch composition of each filler content for tapioca starch-filled HDPE blend.

| Filler Content (%) | The total density of the blend (g) | HDPE (g) | Tapioca starch (g) |
|---|---|---|---|
| 0.5 | 0.7582 | 33.19 | 0.17 |
| 1 | 0.7603 | 33.12 | 0.33 |
| 5 | 0.7778 | 32.51 | 1.71 |
| 10 | 0.8001 | 31.68 | 3.52 |
| 20 | 0.8514 | 29.97 | 7.49 |
| 30 | 0.9088 | 27.99 | 11.20 |

**Table 3.4** Batch composition of each filler content for rice husk-filled HDPE blend.

| Filler Content (%) | The total density of the blend (g) | HDPE (g) | Rice husk (g) |
|---|---|---|---|
| 0.5 | 0.7585 | 33.21 | 0.17 |
| 1 | 0.7610 | 33.15 | 0.33 |
| 5 | 0.7817 | 32.67 | 1.72 |
| 10 | 0.8091 | 32.04 | 3.56 |
| 20 | 0.8702 | 30.63 | 7.66 |
| 30 | 0.9413 | 28.99 | 12.42 |

**Table 3.5** Batch composition of each filler content for burning husk-filled HDPE blend.

| Filler Content (%) | The total density of the blend (g) | HDPE (g) | Burning husk (g) |
|---|---|---|---|
| 0.5 | 0.7583 | 33.20 | 0.17 |
| 1 | 0.7605 | 33.13 | 0.33 |
| 5 | 0.7788 | 32.55 | 1.71 |
| 10 | 0.8031 | 31.80 | 3.53 |
| 20 | 0.8565 | 30.15 | 7.54 |
| 30 | 0.9176 | 28.26 | 12.11 |

The blends obtaining from mixing processes were allowed to cool to room temperature and then ground to small size. The polymer blends were put into a picture frame mold. The mold was preheated at 170 °C for 5 minutes in a Wabash V 50 H Compression Molder and then compressed under a pressure of 10 tons for 3 minutes, followed by cooling to 37 °C under a pressure of 10 tons. Finally, the compressed sheets were cut to desired shape for each testing method according to ASTM standard.

### 3.2.5 Mechanical Properties of Polymer Blends

3.2.5.1 Tensile Properties Testing

Tensile strength and tensile modulus of the blends were measured using an Instron Universal Testing Machine. The test was conducted according to ASTM D 638-91 test procedure.

The specimens were cut in dumbell-shaped form, according to Type I, ASTM D 638-91. Tests were carried out using a 100 kN load cell and 50 mm/min crosshead speed. Five specimens were determined for each sample and the results were averaged to obtain a mean value.

3.2.5.2 Flexural Properties Testing

Flexural properties in term of flexural stress and flexural modulus of the blends were measured using an Instron Universal Testing machine according to Type I, ASTM D 790-92, using the three-point bending method. Measurements were done using a 5 kN load cell, 12mm/min crosshead speed and 46.4 mm of support span. Five specimens were tested for each sample and then the results were averaged to obtain a mean value.

3.2.5.3 Impact Properties Testing

Izod impact strength were measured using a Zwick Impact Test instrument with a 2.7 joules pendulum according to ASTM D 256-90b test method. The five specimens were tested to evaluate a mean value.

### 3.2.6 Water Absorption

Water absorption was measured according to ASTM D 570-81. The test specimens were dried in oven for 24 hours at 50 °C, cooled in a desiccator, and then weighed. After that, the test specimens were immersed in distilled water. At regular time intervals, specimens were removed, wiped dry with blotting paper, and then weighed to determine the water uptake. The specimens were placed back in the water after each measurement. The water absorption was calculated as the weight difference and reported as the percent increase of the initial weight. The specimens were done in triplicate and the results were reported as an averaged value of the three measurements.

### 3.2.7 Scanning Electron Microscope

Fracture surface of the specimen obtaining from impact testing were investigated with a scanning electron microscope (SEM), using a JEOL microscope model JSM 5200. The specimens were coated with gold and observed by SEM. A Semafore program was used to measure sizes of the fillers in polymer blends.