

## CHAPTER II

### EXPERIMENTAL SECTION

#### 2.1 Materials

##### 2.1.1 Starch

Tapioca starch and rice starch used in this study were kindly supplied by Siam Modified Starch Co., Ltd. The properties of tapioca and rice starch are given in Table 2.1

**Table 2.1** Properties of tapioca starch and rice starch

Properties	Tapioca starch	Rice starch
Particle size	10 – 15 $\mu\text{m}$	3 – 5 $\mu\text{m}$
Particle shape	spherical	polygonal

##### 2.1.2 High density polyethylene

High density polyethylene (HDPE) film extrusion grade A3355 obtained from Thai Petrochemical Industry Co., Ltd. was used as the polymer matrix. The melt flow index of HDPE was 0.06 g/10 min.

### 2.1.3 Natural rubber

Natural rubber, supplied by Victor & Prosper Limited Partnership, was used as an autooxidant to promote the oxidation reaction of HDPE.

### 2.1.4 Zinc stearate

Zinc stearate, obtained from Imperial Industrial Chemicals (Thailand) Co., Ltd., was used as a metal catalyst to catalyze the oxidative degradation of HDPE. The melting point of zinc stearate was 120-124°C.

### 2.1.5 Calcium oxide

Calcium oxide (CaO) was used as a desiccant of the starch masterbatch.

## 2.2 Experimental Procedures

### 2.2.1 Masterbatch Preparation

*2.2.1.1 Starch Masterbatch.* A masterbatch containing 50% starch was prepared in a twin screw extruder (Collin ZX-25). The starch was dried in a hot-air oven at 100°C for 2.5 hr before use. 50 wt % of dried starch, 4 wt % of calcium oxide and 46 wt % of HDPE were premixed in a tumble mixer and subsequently fed into the hopper of the extruder. The melt was extruded through a single strand die, air cooled and pelletized. The processing conditions of the starch masterbatch are shown in Table 2.2.

**Table 2.2** Processing conditions of the starch masterbatch

Extrusion parameter	Operating value
Extruder temperature profile (°C) :	
zone 1	145
zone 2	155
zone 3	160
zone 4	160
zone 5	160
die zone	160
Screw speed (rpm)	55
Feed rate (arbitrary units)	200

*2.2.1.2 Zinc Stearate Masterbatch.* 10 wt % of zinc stearate and 90 wt % of HDPE were tumble mixed before blending in a twin screw extruder (Collin ZX-25). The processing conditions of the zinc stearate masterbatch are shown in Table 2.3.

*2.2.1.3 Rubber Masterbatch.* A blend of 25 wt % natural rubber and 75 wt % HDPE was compounded on a Lab Tech LRM 110 two roll mill. The temperatures of the front roll and the back roll were 160°C and 150°C, respectively. The masterbatch was processed for 8 minutes after which it was removed from the mill and comminuted in a granulator.

**Table 2.3** Processing conditions of the zinc stearate masterbatch

Extrusion parameter	Operating value
Extruder temperature profile (°C) :	
zone 1	110
zone 2	120
zone 3	145
zone 4	160
zone 5	160
die zone	160
Screw speed (rpm)	40
Feed rate (arbitrary units)	200

### 2.2.2 Sample Preparation

Starch-based HDPE blends were prepared in a twin screw extruder (Collin ZX-25). Appropriate amounts of HDPE and starch masterbatch were blended together to give HDPE/starch blends with 5%, 10%, 15%, 20%, 25%, 30%, 35% and 40% starch by weight. When rubber and zinc stearate were incorporated they were added at levels of 2 wt % and 1wt %, respectively. These values were fixed for all composition formulas.

The formulations were premixed by tumble mixing and subsequently fed into the hopper of the extruder. The melt was extruded through a single strand die, air cooled and pelletized. Pellets were kept in sealed plastic bags until molding to minimize moisture regain from the

atmosphere. The processing conditions of starch-based HDPE blends are shown in Table 2.4.

**Table 2.4** Processing conditions of starch-based HDPE blends

Extrusion parameter	Operating value
Extruder temperature profile (°C) :	
zone 1	145
zone 2	155
zone 3	160
zone 4	160
zone 5	160
die zone	160
Screw speed (rpm)	55
Feed rate (arbitrary units)	300

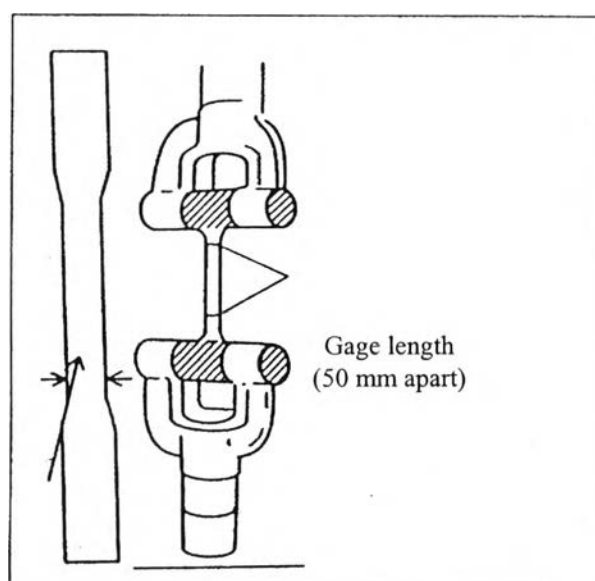
### 2.3 Mechanical Testing

The samples for mechanical testing were prepared from compression molded sheet using a Wabash V 50 H compression molder. The pellets were preheated at 180 °C for 5 minutes and then were compression molded in a picture frame mold under a force of 5 tons for 5 minutes followed by a force of

20 tons for 5 minutes. The compression molded sheet was then cooled down to 50 °C.

### 2.3.1 Tensile Testing

Tensile properties were measured using an Instron Universal Testing Machine, Model 4206, according to the ASTM D638 test procedure. The diagram of a tensile test is shown in Figure 2.1.

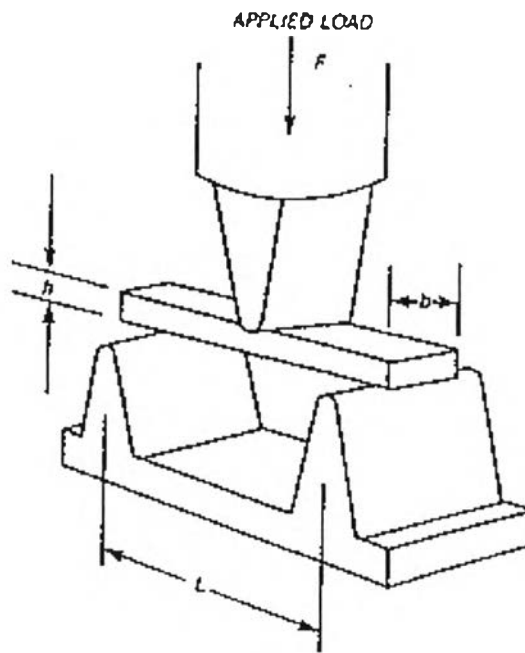


**Figure 2.1** The diagram of a tensile test, showing the dumbbell-shaped specimen (Type I, ASTM D638) clamped in the jaws of an Instron tester.

Tests were carried out using a 5 kN load cell and 50 mm/min crosshead speed. Type I dumb-bell specimens were prepared by die cutting. Five specimens were tested for each sample and the results were averaged to obtain a mean value.

### 2.3.2 Flexural Testing

Flexural properties were measured using an Instron Universal Testing Machine, Model 4206, according to the ASTM D790-92 type I, using the three-point bending method as shown in Figure 2.2



Where  $h$  = the thickness of the specimen  
 $b$  = the width of the specimen  
 $L$  = the length of the support span

**Figure 2.2** 3 - point bending for flexural testing.

Measurements were made using a 5 kN load cell, 13 mm/min crosshead speed and 46.8 mm support span. The dimension of the test specimens were 12.75 mm x 127.5 mm x 3.00 (nominal) thickness. Five

specimens were tested for each sample and the results were averaged to obtain a mean value.

### 2.3.3 Izod Impact Testing

Izod impact strengths were determined on V-notched specimens using a Zwick Impact Test instrument according to the ASTM D256-90b test method using a 2.7 joules pendulum. The dimension of the test specimens were 12.7 mm x 63.5 mm x 3.00 mm (nominal) thickness. Each specimen was machined to produce a V-notch having a 45 degree angle and 2.5 mm depth.

## 2.4 Scanning Electron Microscope

The fracture surfaces were examined by secondary electron images, using a JEOL JSM 5200 scanning electron microscope (SEM). Prior to examination, the fracture surface was coated with a thin evaporated layer of gold in order to improve conductivity and prevent electron charging on the fracture surface.