

CHAPTER III

EXPERIMENTAL SECTION

3.1 Materials

3.1.1 High Density Polyethylene (HDPE)

The HDPE used in this study was blown-film grade Thai-zex 7000F, provided by Bangkok Polyethylene Co., Ltd. Some physical properties of the HDPE are shown in table 3.1

Table 3.1 Physical properties of HDPE

Properties	Unit	Test method	Typical values
Melt flow rate	g/10min	ASTM D1238	0.04
Density	g/cm ³	ASTM D1505	0.956
Tensile strength at yield	Kg/cm ²	ASTM D 638	280
Elongation at break	%	ASTM D 638	>500
Izod impact strength	Kg.cm/cm ²	ASTM D 256	30
Hardness (Shore D)	D Scale	ASTM D 2240	64
Melting point	°C	ASTM D 2117	131
Softening point	°C	ASTM D 1525	124

3.1.2 Starch

Tapioca starch used in this study was kindly supplied by Siam Modified Starch Co., Ltd.

3.1.3 Compatibilizers

3.1.3.1 *Poly(ethylene-co-acrylic acid): EAA*

EAA copolymers containing 20 wt% acrylic acid was purchased from Aldrich Chemical Company, Inc.

3.1.3.2 *Poly(ethylene-co-vinyl acetate); EVA*

EVA copolymers containing 18 wt% vinyl acetate was purchased from Aldrich Chemical Company, Inc.

3.1.3.3 *Poly(ethylene-graft- maleic anhydride); PE-g-MA*

PE-g-MA copolymers containing 0.8 wt% anhydride group was purchased from Aldrich Chemical Company, Inc.

3.1.4 Diethyl ether

Diethyl ether ($C_2H_5OC_2H_5$) was used as non-solvent for measuring specific gravity of tapioca starch by the pycnometric technique.

3.2 Experimental Procedure

3.2.1 Starch preparation

Tapioca starch was dried for 3 hours in a hot-air oven at 110°C to reduce the moisture content and then kept in sealed containers until used.

3.2.2 Thermogravimetric analysis of starch

In order to determine the decomposition temperature and the water content of tapioca starch used in this study, the thermogravimetric analysis was conducted by heating the starch from 50°C to 600°C at a heating rate of 10°C/min using a Perkin Elmer TGA7 analyzer.

3.2.3 Starch density measurement

The density of starch was measured using the pycnometric technique. The method involves the use of a pycnometer of known volume. The starch to be evaluated was weighed and put into the pycnometer. The pycnometer containing starch was then filled with diethyl ether of known density and reweighed. The weight and thus the volume of diethyl ether could then be

found from the weight difference. The density of the starch could then be determined from the following equation:

$$\rho_{st} = [W_{st} / (V_{pyc} - (W_{ether} / \rho_{ether}))] \quad (3.1)$$

where ρ_{st} is the density of the starch (g/cm^3)
 W_{st} is the weight of the starch contained in the pycnometer (g)
 V_{pyc} is the volume of the pycnometer = 25.261 cm^3
 W_{ether} is the weight of the diethyl ether contained in the pycnometer (g)
 ρ_{ether} is the density of the diethyl ether = $0.7107 \text{ g}/\text{cm}^3$

The value of starch density measured by pycnometric technique was used to calculate the density of the blends and then the batch size of mixing.

3.2.4 Polymer blends preparation

HDPE/starch blends were prepared by using a Brabender Plastimeter, PL-2100, equipped with a cam-type mixer-measuring head. The mixer-measuring head consists of an interconnected, figure-eight-shaped chamber, having a capacity of 80 cm^3 , in which two sigmoid, counter-rotating blades turn. A rotor speed of 60 rpm and an oil bath temperature of 170°C were used. All runs were carried out on by using Shear Stability mode and a fill factor of 0.85. Starch contents were varied from 0-40 wt%. Compatibilizers were added by vary from 0-40 wt% based on starch content in the polymer blends. An average value of HDPE melt density was $0.756 \text{ g}/\text{cm}^3$ while an average melt density of each compatibilizer used was $0.7 \text{ g}/\text{cm}^3$. Batch sizes were calculated from the following equation:

$$\text{Batch size (g)} = V_c \times D_b \times F \quad (3.2)$$

where V_c is Volume of chamber (cm^3)

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

F is Fill factor

The components were physically premixed before being fed into the mixer-measuring head. The torque required to turn the blades at the set rotation speed was transmitted from the dynamometer housing to the computer to record the torque value as a function of time. The stock temperature was measured by a thermocouple positioned inside the chamber. After mixing, the blends were then taken off, milled in a shredder and placed in tightly sealed containers to prevent any moisture absorption.

3.3 Mechanical Testing

The samples for mechanical testing were prepared from compression molded sheet using a Wabash V 50H compression molder. The mold used in this study was a 12.7 x 16.5 cm picture frame stainless steel mold of thickness 3 mm. The shredded HDPE/starch blends were preheated at 170°C for 3.5 minutes and then were compressed under a force of 10 tons for 30 seconds before cooled down to 35°C. Finally, specimens were press-cut by Pneumatic Specimen Cutter for further mechanical testing.

3.3.1 Tensile testing

Tensile properties were measured by using an Instron Universal Testing Machine, Model 4206, according to the ASTM D 638 test procedure. Measurements were carried out using a 5 kN load cell and 50 mm/min crosshead speed. Type I specimens were used in which the width of the narrow section was 13 mm, the length of narrow section was 57 mm with

gauge length of 50 mm. Five specimens were tested for each sample and the results were averaged to obtain a mean value.

3.3.2 Flexural testing

Flexural properties were measured by using an Instron Universal Testing Machine, Model 4206, according to the ASTM D 790-92 type I using the three-point bending method. Measurements were performed using a 5 kN load cell, 12.8 mm/min crosshead speed and 48 mm support span. The dimension of the test specimens was 12.0 mm x 80.0 mm x 3.0 mm (nominal) thickness. Five specimens were tested for each sample and the results were averaged to obtain a mean value.

3.3.3 Izod impact testing

Izod impact resistances were determined on V-notched specimens using a Zwick Impact Test Instrument, Model 5113, according to the ASTM D 256-90b test method using a 2.7 joules pendulum. The dimension of the test specimens was 12.7 mm x 63.5 mm x 3.0 mm (nominal) thickness. Each specimen was machined to produce a V-notch having a 45 degree angle and 2.5 mm depth.

3.4 Microstructure Characterization

The morphology of the HDPE/starch blends was examined by secondary electron images, using a JEOL scanning electron microscope (SEM) Model JSM 5200. The operating voltages were in the range of 1-10 kV. The fractured samples obtained after impact testing were cut about 2 mm below the fractured surface and stuck to aluminium stubs. Prior to examination, the surface of the specimen was coated with a thin evaporated layer of gold under vacuum for 3 minutes in order to improve conductivity and

prevent electron charging on the surface. SEM micrographs of the samples were taken at magnifications ranging from 200 to 500.

3.5 Water Absorption Measurement

Water absorption of the blends was determined according to ASTM D 570-81. The dimension of the test specimen was 76.2 mm x 25.4 mm x 3.0 mm (nominal) thickness. The test specimens were conditioned by drying in an oven for 24 hours at 50°C and then cooled down in a desiccator. The weights of the specimens were recorded before immersing them into the water bath containing distilled water maintained a temperature at $23\pm 1^\circ\text{C}$. The specimens were then removed from water bath at specified time intervals. Surfaces of the specimens were wiped off with blotting papers and the weights were recorded. The specimens were placed back into the distilled water after each measurement. The water absorption was calculated as the weight difference and reported as the percent increase of the initial weight. Three specimens of each sample were tested and the results were reported as an average value.