

## **CHAPTER III**

### **EXPERIMENTAL**

#### **3.1 Materials**

##### **3.1.1 High-Density Polyethylene**

Two types of reactor-grade high-density polyethylene were used. One was supplied by Thai Polyethylene Co. Ltd., (TPE), H5690S with a melt flow index (MFI) of 0.9 g/10 min (190°C, 2.16 kg) , narrow molecular weight distribution, molecular weight more than  $2 \times 10^4$  ,and a density of 0.954 g/cm<sup>3</sup>. The second was supplied by Bangkok Polyethylene Co. Ltd., 5000s, with a melt flow index (MFI) of 0.8 g/10 min (190°C, 2.16 kg) and a density of 0.954 g/cm<sup>3</sup>.

##### **3.1.2 Antioxidants**

Antioxidants from tow different sources were employed in the study. First was the Irganox series, Irganox 1010 primary antioxidant and Irganos 168 secondary antioxidant from Ciba Specialty Chemical, Switzerland and second was the Reonox series, Reonox 10 primary antioxidant and Reonox 68 secondary antioxidant, from Eonian Co., Ltd., Taiwan.

#### **3.2 Processing**

##### **3.2.1 Preparation of Antioxidant Masterbatches**

Reactor-grade HDPE from TPE (type H5690S) and BPE (type 5000s) were premixed with primary and secondary antioxidants at 10% by wt in BOSCO tumble mill for 10 minutes. After that mixed material was compounded by COLLIN co-rotating twin screws extruder KNEADER ZK-25

(25mm × 30D) and masterbatches extrudate was pelletized by Planetrol 075D2 pelletizer. The amount of materials prepared were presented in Table 3.1.

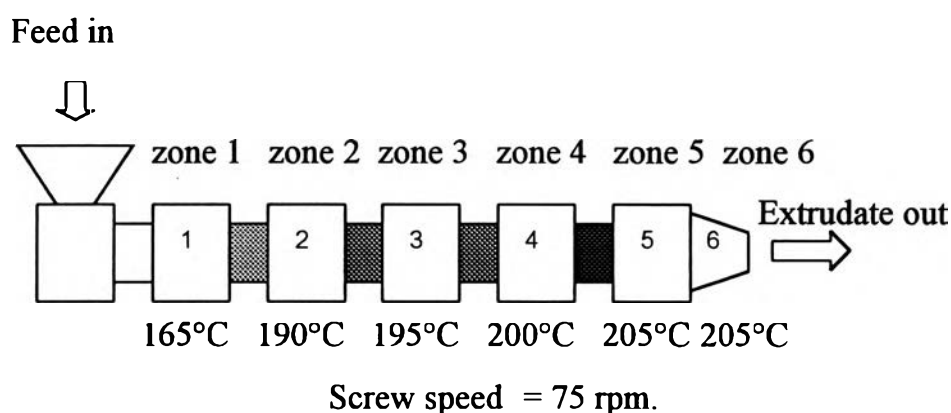
**Table 3.1** Masterbatches preparations at 10% by wt antioxidants content.

	TPE			
	Irganox 1010	Irganox 168	Reonox 10	Reonox 68
Amount of HDPE (g)	450	450	450	450
Amount of antioxidant (g)	50	50	50	50
Total (g)	500	500	500	500

	BPE			
	Irganox 1010	Irganox 168	Reonox 10	Reonox 68
Amount of HDPE (g)	450	450	450	450
Amount of antioxidant (g)	50	50	50	50
Total (g)	500	500	500	500

### 3.2.2 Compounding of HDPE with Antioxidant Masterbatch

Preparation methods of stabilized HDPE were similar to masterbatches preparation steps but substitute 10% by wt. antioxidant by 10% by wt. masterbatches, as represented in Table 3.2. All processing passes were carried out with the following conditions as presented in Fig. 3.1.



**Figure 3.1** Processing conditions of twin screw extruder.

**Table 3.2** The compositions of stabilized and non-stabilized HDPE.

Compounding Sample (1° antioxidant/2° antioxidant)	Amount of polymer	Amount of antioxidants	
	HDPE (g)	Primary antioxidant (g)	Secondary antioxidant (g)
Virgin HDPE <sup>1</sup> (0/0)	1200	-	-
Virgin HDPE <sup>2</sup> (0/0)	1200	-	-
HDPE <sup>2</sup> + Irganox 1010 (100/0)	1200	12.12	-
HDPE <sup>1</sup> + Irganox 1010 (100/0)	1200	12.12	-
HDPE <sup>1</sup> + Irgafos 168 (0/100)	1200	-	12.12
HDPE <sup>2</sup> + Irgafos 168 (0/100)	1200	-	12.12
HDPE <sup>1</sup> + Irganox 1010/Irgafos 168 (25/75)	1200	3.03	9.09
HDPE <sup>2</sup> + Irganox 1010/Irgafos 168 (25/75)	1200	3.03	9.09
HDPE <sup>1</sup> + Irganox 1010/Irgafos 168 (50/50)	1200	6.06	6.06
HDPE <sup>2</sup> + Irganox 1010/Irgafos 168 (50/50)	1200	6.06	6.06

**Table 3.2 (Continued)**

Compounding	Amount of polymer	Amount of antioxidants	
		Primary antioxidant	Secondary antioxidant
Sample (1° antioxidant/2° antioxidant)	HDPE  (g)	Primary antioxidant  (g)	Secondary antioxidant  (g)
HDPE <sup>1</sup> + Irganox 1010/Irgafos 168 (75/25)	1200	9.09	3.03
HDPE <sup>2</sup> + Irganox 1010/Irgafos 168 (75/25)	1200	9.09	3.03
HDPE <sup>2</sup> + Reonox10 (100/0)	1200	12.12	-
HDPE <sup>1</sup> + Reonox 10 (100/0)	1200	12.12	-
HDPE <sup>1</sup> + Reonox 68 (0/100)	1200	-	12.12
HDPE <sup>2</sup> + Reonox 68 (0/100)	1200	-	12.12
HDPE <sup>1</sup> + Reonox 10/Reonox 68 (25/75)	1200	3.03	9.09
HDPE <sup>2</sup> + Reonox 10/Reonox 68 (25/75)	1200	3.03	9.09
HDPE <sup>1</sup> + Reonox 10/Reonox 68 (50/50)	1200	6.06	6.06
HDPE <sup>2</sup> + Reonox 10/Reonox 68 (50/50)	1200	6.06	6.06
HDPE <sup>1</sup> + Reonox 10/Reonox 68 (75/25)	1200	9.09	3.03
HDPE <sup>2</sup> + Reonox 10/Reonox 68 (75/25)	1200	9.09	3.03

HDPE<sup>1</sup> = HDPE supplied by TPE

HDPE<sup>2</sup> = HDPE supplied by BPE

The extrudate of compounded HDPE was cooled in water at temperature approximately 27°C and pelletized by Planetrol 075D2 pelletizer. In case of study the effect of antioxidants on processability (ability of recycle), the compounded HDPE was reprocessed again by recycle compounded HDPE into twin screw extruder. It was done until number of passes reach 7 times.

### **3.3 Sample Preparation**

Sample preparation was divided into 3 parts :

1. Sample for melt flow index (MFI) testing was obtained from sample pellets of each sample.
2. Specimens for mechanical testing were prepared from sheets, which were molded using Wabash compression molding machine. The 60g. of pellets were placed into a picture frame mold and preheated at 170 °C for 3 minutes. After preheating, forces of 10 tones was applied on the sample for 3 minutes. Then sample sheet was cooled until temperature approach to ambient temperature. Dumbbell shape cutter was used to dumbbell shape samples.
3. Sample film for FTIR analysis, It was prepared by compression molding. A little amount of sample pellets was compressed within flat and smooth stainless steel plate. A 100 µm average thickness of sample film was determined using a PEACOCK PDP-1 thickness gauge.

### **3.4 Aging Procedure**

Pellet samples, dumbbell samples and sample for impact resistance were aged in a hot air oven. Thin cotton thread wires were used to hang dumbbell shape samples. The pellet shape samples were placed in the container, which made from a sieve. Oven temperature was maintained at 105°C ± 5°C, simultaneously fresh air was fed for all aging duration. All

recipes were removed from oven every 2, 4, 8, 16, 32 and 64 days. Samples at various sampling times were used for physical properties testing i.e. MFI, tensile property and impact resistance. Functional groups analysis was also carried out on the removed samples to detect for any functional groups that could indicate signs of degradation (e.g. CO or OH and more).

### **3.5 Testing Procedure**

#### **3.5.1 Melt Flow Index (MFI)**

Melt flow index of all samples were determined according to ASTM D 1238 by Zwick 405 Extrusion Plastometer with piston load weight of 2.16 kg at 190°C. Both melted stabilized HDPE and non-stabilized HDPE were past through the die which has diameter of 1.180 mm. For a required cutting time, HDPE extrudate were weighing and scale up or scale down to the unit of g/10 minutes depend on cutting time that set on Zwick 405 Extrusion Plastomer. Zwick 4.5 Extrusion Plastometer was also employed in the study of processability of the different HDPE samples.

#### **3.5.2 Tensile Test**

Tensile test was one of the many mechanical property tests. Dumbell-shaped samples were die cut from sheets. Specimens were drawn in an Instron Universal testing machine at the constant cross-head speed of 50 mm/min. The test was carried out according to ASTM D 638-91.

#### **3.5.3 Differential Scanning Calorimetry (DSC)**

Differential Scanning Calorimetry was used to determine the OIT of the polymeric materials. Oxidative induction time was carried out on a Netsch DSC 200. Samples of 3-10 mg were placed in open aluminum pans. The temperature was programmed at a heating rate of 20°C/min. from ambient

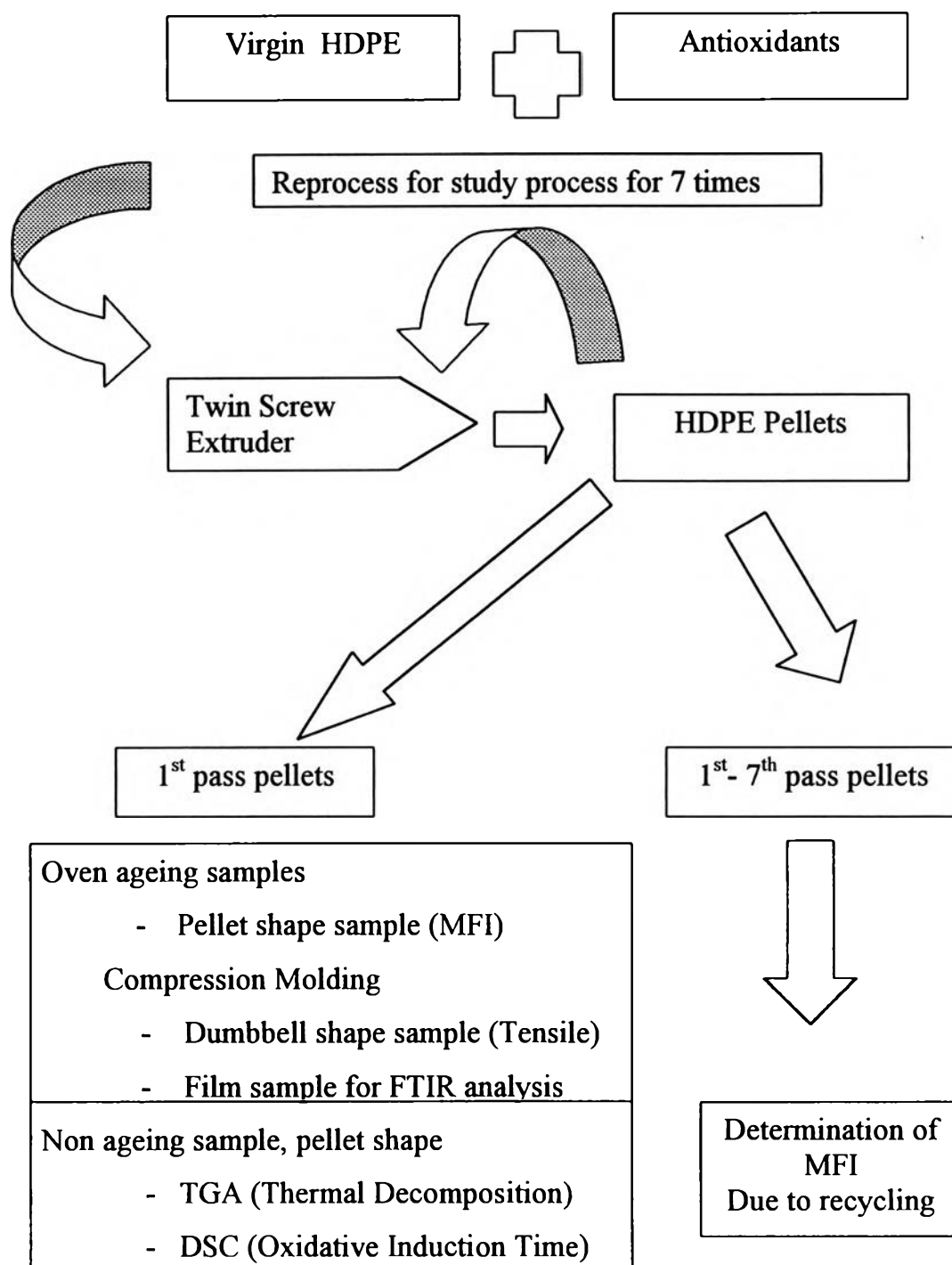
temperature to 200°C. The chamber was purged with dry nitrogen gas at flow rate of 25 ml/min until temperature had reached 200°C. This system was programmed for 5 minutes equilibrate. The purged nitrogen gas was then quickly changed to oxygen. The specimen was then held at a constant temperature until the oxidative reaction was displayed on the thermogram. This test was carried out according to ASTM D 3895-92.

#### 3.5.4 Thermo Gravimetric Analysis (TGA)

3-10 mg of stabilize HDPE pellet samples were used to measure volatility in Perkin Elmer DSC 7. The samples were heated from ambient temperature to 500°C at heating rate of 20°C/min to obtained thermogram or thermal decomposition of each recipe.

#### 3.5.5 Fourier Transform Spectroscopy Analysis (FTIR)

Fourier Transform Spectroscopy analysis, Films of both aging and non-aging HDPE samples were used FTIR analysis. The film samples was use in the analysis was 100µm thick. The FTIR analysis used to determine the functional groups was carried out on BRUKER EQUINOX55/S, MIR, FTIR-Source with 16 numbers of scans. All experimental procedures have been explained shortly by Figure. 3.2.



**Figure 3.2** Measuring the effect of antioxidants on thermal degradation of HDPE.