Chapter III

Experiment

3.1 Materials

3.1.1 Medium Density Polyethylene

Medium density polyethylene (MDPE), rotational molding grade (M3804 RWP/RW), used in this investigation was supplied by Thai Polyethylene Co., Ltd.. Thailand under the trademarks MDPE EL-Lene. MDPE EL-Lene was derived from the gas phase process of BP Chemical Co., Ltd., England. The properties of the MDPE EL-Lene used are summarized in Table 3.1.

Table 3.1 Characteristics of the MDPE EL-Lene [52]

Properties	Unit	Testing method	Typical values
Melt Flow Rate	g/10 min	ASTM D 1238	4.0
Density	g/cm^3	ASTM D 1505	0.938
Tensile Strength at Yield	kg/cm^2	ASTM D 638	200
Tensile Strength at Break	kg/cm^2	ASTM D 638	270
Elongation at Break	%	ASTM D 638	1,000
Flexural Modulus	kg/cm^2	ASTM D 790	6,700
Izod Impact Strength	Kg-cm/cm	ASTM D 256	13
Hardness, shore D	-	ASTM D 2240	60
Environmental Stress Cracking Resistance	F_{50}, hr.	ASTM D 1693	> 200
Melting Point	°C	ASTM D 2117	128
Vicat Softening Point	°C	ASTM D 1525	117
Brittleness Temperature	°C	ASTM D 746	< -70
Average Partical Size	Micron	TPE Method	350
UV Resistance	KLy.	ASTM D 2565	500

3.1.2 Organic Pigment

The organic pigments used in this study were obtained from Drycolor Pacific Ltd., affiliated company of Clariant. Three organic pigments, including phthlocyanine, diarylide, and quinacridone, were studied. Their color index names are PB15 (blue), PY83 (yellow), and PR122 (red), respectively. Table 3.2 presents the characteristics of each pigment.

Table 3.2 Characteristics of phthalocyanine pigment (PB15), diarylide pigment (PY83), and quinacridone pigment (PR122) [53 - 55]

Properties	Pigments		
	PB15	PY83	PR122
Density (g/cm^3)	1.63	1.49	1.47
Bulk Density (l/kg)	3.5	4.0	3.2
Average Particle Size (nm)	40	55	95
Specific Surface (m^2/g)	64	80	65
Hue Angle (^{o})	248.2	79.9	338.8
Chroma	46.8	76.0	55.2
Heat Resistance (^{o}C)	300	200	300

3.2 Instruments

Details of each instrument are classified according to the experimental procedure as follows. Appendix A shows pictures of each instrument.

3.2.1 Sample Preparation

1) Dry Blender

A Red Devil 5400/5410 Paint Mixer was used to mix MDPE powder and pigments for dry blending technique.

2) Twin Screw Extruder

The compound process of mixture was done by twin screw extruder, model TSE systems of Thermo Prism, for melt blending technique.

3) Compression Molding

Compression molding machine was used to prepare the plastic sheet from both dry mixture and pelletized extrudate of MDPE and organic pigments.

4) Cutter

Cutter was used to change the plastic sheets into the rectangular shape for flexural and impact tests specimens.

5) Lathe

The dumbbell specimens for tensile testing were prepared by the lathe with contour holder.

3.2.2 Mechanical Property Testings

1) Tensile Testing

Tensile properties of the specimen were tested according to the ASTM D 638 type IV using universal testing machine.

2) Flexural Testing

Similar to tensile test, universal testing machine was employed to measure the flexural properties of the specimen according to the ASTM D 790.

3) Impact Testing

Izod-impact tests of the plastic specimens were performed by izod impact tester according to the ASTM D 256 (Izod-Type).

3.2.3 Physical Property Characterization

1) Scanning Electron Microscope (SEM)

Scanning electron microscope was used to observe the fractured surface of the samples.

2) Melt Flow Index Tester

The melt flow index tester was employed to measure the melt flow index (MFI) of the plastic samples in accordance with the ASTM D 1238.

3) Laser Particle Size Analyzer

Particle size of the individual organic pigments and MDPE were carried out by a laser particle size analyzer.

3.2.4 Thermal Property Characterization

Differential Scanning Calorimetry (DSC)

Thermal properties of plastic samples were analyzed by differential scanning calorimetry.

The details of each instrument are tabulated in Table 3.3 and their features are exhibited in Appendix A.

Table 3.3 Model and manufacturer of the instruments used in this research

	Instrument	Model	Manufacturer
Sample Preparation	Dry Blender	Red Devil 5400/5410 Paint Mixer	Red Devil
	Twin Screw Extruder	TSE Systems	Thermo Prism
	Compression Molding	4 Hydraulic Column	-
	Cutter	-	Yasuda Seiki Seisakusho Ltd.
	Lathe	-	ATS FAAR SPA MILANO / ITALIA
	Izod Impact Notcher	Yasuda Seiki No.189-PFN	Yasuda Seiki Seisakusho Ltd.
Mechanical Property Characterization	Tensile Testing Machine	Universal Testing Machine LLOYD LR 100K	LLOYD Instruments
	Flexural Testing Machine	Universal Testing Machine LLOYD LR 100K	LLOYD Instruments
	Izod Impact Tester	Yasuda Seiki No.258-D	Yasuda Seiki Seisakusho Ltd.
Physical Property Characterization	Scanning Electron Microscope	JSM – 5410 LV Scanning Microscope	JEOL
	Melt Flow Index Tester	7053	KAYENESS INC.
	Laser Particle Size Analyzer	Mastersizer S long bed Ver. 2.11 Serial Number : 32734 - 89	Malvern Instruments Ltd.
Thermal Property Characterization	Differential Scanning Calorimetry	DSC 822^e	Mettler Toledo Ltd.

3.3 Methodology

Flow chart of the experimental process is shown below in Figure 3.1.

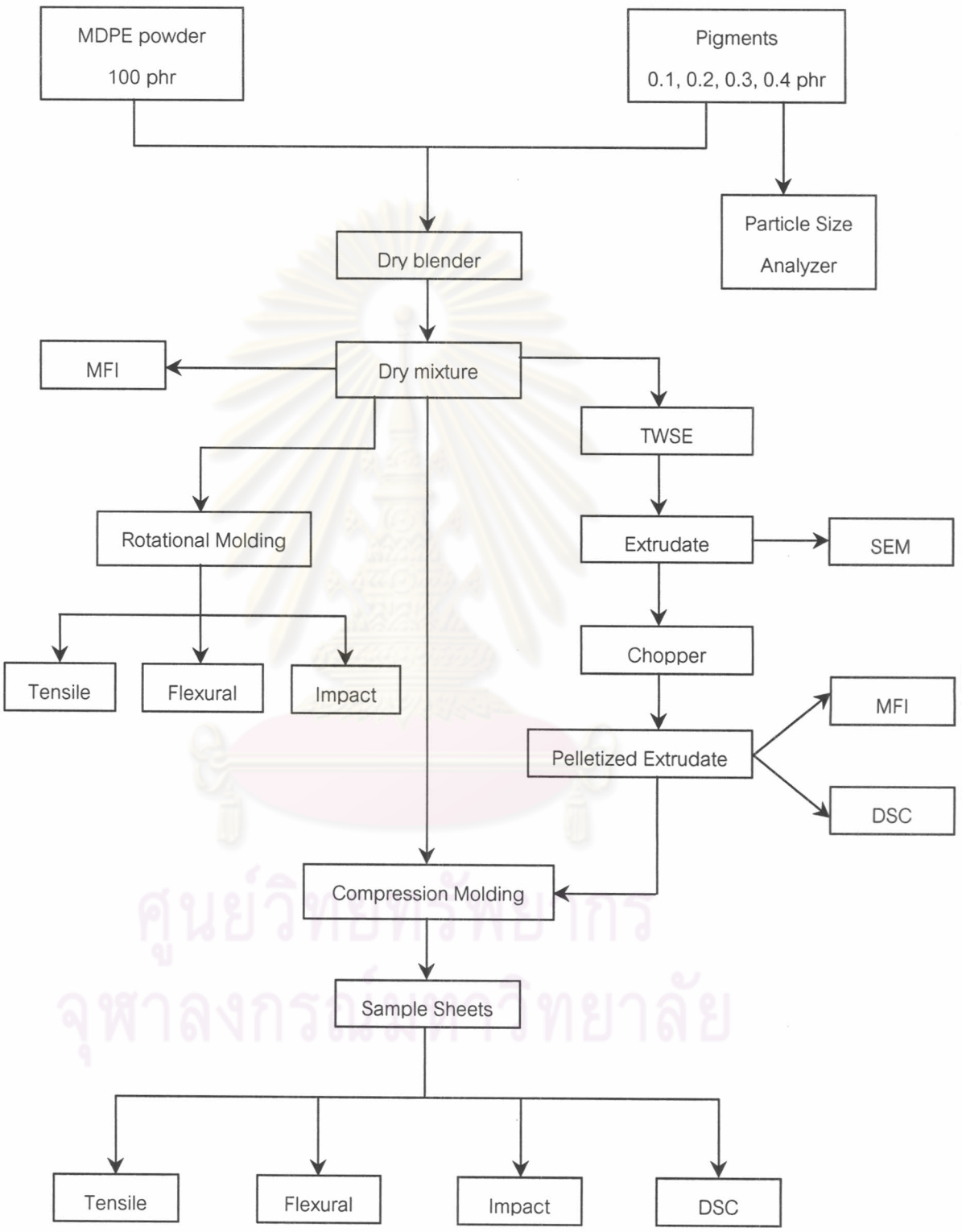

Figure 3.1 The flow chart of experimental procedure

3.3.1 Blending Technique

For comparative studies, two blending techniques, melt and dry blending techniques, were used in this research. The ratio and amount of each component, MDPE and pigment powder, for both techniques are summarized in Table 3.4.

Table 3.4 Content of mixture between medium density polyethylene (MDPE) and pigment powder

Material Powder	Proportion (phr)	Weight in Can / Trial (g)
MDPE	100	600
Organic Pigments	0.1	0.6
	0.2	1.2
	0.3	1.8
	0.4	2.4

3.3.1.1 Dry Blending Technique

For dry blending technique, the MDPE and pigments were mixed in the cans. The amount of mixture in the can, should be at least up to 2 in 3 of its volume for good dispersibility. The cans were shaken up on the dry blender for 30 minutes per batch. Finally, the output was dry mixture.

3.3.1.2 Melt Blending Technique

The mixture from dry blending technique was extruded through a strand die and subsequently pelletized by a twin screw extruder. Prior to doing so, the suitable blending condition for twin screw extruder such as screw speed and processing temperature has to be chosen. Table 3.5 lists various blending conditions for twin screw extruder. The consideration was mainly based on the tensile properties and processing time which will be discussed in Chapter IV.

The quinacridone pigment (PR122) was selected as the representative of the pigments used in this research. The amount of PR122 was 2.4 g (0.4 phr) per 1000 g of MDPE powder for every blending condition. After the appropriate condition was found, the melt blending of the MDPE powder and three organic pigment powder was performed using the composition summarized in Table 3.4.

Table 3.5 The blending conditions in twin screw extruder

Condition No.	Screw Speed (Rotational Speed of Screw) (rpm)	Processing Temperature (Mixing Temperature) (°C)
1	10	160
2		180
3		200
4	20	160
5		180
6		200
7	30	160
8		180
9		200

3.3.2 Sample Sheet Preparation

Compression molding was used to prepare the sample sheet of 9" wide by 9" long and 3 mm thick. Approximately 85 g of the mixture from either blending technique was pressed between two plates of the mold under pressure of 5 MPa at 135 °C for 10 min. After slow cooling the sample sheet to the ambient temperature for about 3 hours in the air, the specimens were prepared for the mechanical properties testing as follows.

The sample sheets for tensile testing were cut into a rectangular shape (19 mm X 115 mm) using a cutter. They were then turned by the lathe to the dumbbell shaped specimens with the width of narrow section of about 6 mm.

The preparative procedure for the flexural test specimens is also similar to the tensile testing. The sample sheets were cut by a cutter into a rectangular shaped specimen of 12.7 mm wide and 127 mm long.

For impact testing, the cutter was used to cut the sample sheets into the size of 64 X 12.7 $(mm)^2$. The specimens were then notched by a notcher into V-shaped of 2.54 mm depth.

3.4 Characterization and Testing

3.4.1 Mechanical Properties

Tensile testing was carried out at the ambient temperature according to the ASTM D 638 specifications on the Universal Testing Machine, model LLOYD LR 100K. During the tensile test, standard specimens were deformed at a constant crosshead speed of 50.0 mm min^{-1} with an initial gauge length of 25.0 mm. The general purpose in mode of pull to break setup with 10 kN of load cell was used in this test. For flexural testing, the specimens were also tested on the Universal testing machine followed the ASTM D 790. Using the three point bending mode, preload of 5 N was used and the specimens were tested using 1000 N of load cell. The span length was 100 mm and the specimen was compressed with the speed of 1 mm sec^{-1}. Izod-impact testing was performed using the ASTM D 256.

At least five specimens of each sample were tested and the results were averaged to obtain a mean value. Sizes and dimensions of the specimens for each test were mentioned previously in section 3.3.2.

3.4.2 Physical Properties

Scanning electron microscope (SEM) was used to study the morphology of MDPE/pigment mixtures. The extrudate from twin screw extruder was submerged into liquid nitrogen for approximately 5 minutes, depending on the toughness of MDPE, and then fractured. The fractured specimens were then sputter coated with gold to enhance their conductivity and scanned at an accelerating voltage of 15 kV.

The rheological property of MDPE/pigment mixtures from both dry and melt blending techniques was studied by a melt flow index tester, model 7053 of Kayeness Inc. The melt flow index (MFI) was evaluated at 190 $^{\circ}C$ and 2.16 kg weight, following the ASTM D 1238. At least five specimens were tested to obtain the average value.

Particle size of the three organic pigments was measured by the laser particle size analyzer. Water was used as the dispersing medium.

3.4.3 Thermal Properties

The assessment of a polymer's thermal properties can be most easily performed using differential scanning calorimetry (DSC) which measures the heat flow into or from a sample as it is either heated, cooled or under isothermally [56]. These properties of MDPE/pigments component sheets were carried out on a DSC 822^e apparatus, Mettler Toledo Ltd. Hermitically sealed aluminium pans were used to encapsulate the sample of approximately 3 – 4 mg. The sample was loaded into the DSC pan at the ambient temperature and then heated at 10 °C min^{-1} under nitrogen atmosphere (60 ml min^{-1}) to 160 °C. Melting temperature (T_m) and heat of fusion (ΔH_f) of the samples were analyzed from the first heating scan. The reference heat of melting for 100% crystalline PE is 293.6 J/g. The percent crystallinity is then determined using the following equation [56, 57] :

$$\chi_c = \frac{\Delta H_f}{\Delta H_f^c} \times 100$$

Where χ_c is the percent crystallinity,

ΔH_f is the enthalpy of fusion (J/g), and

ΔH_f^c is the enthalpy of fusion of theoretical 100% crystalline polymer (J/g).