CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 High-density Polyethylene (HDPE)

High-density polyethylene employed in this study was l-HDPE and h-HDPE supplied by SCG Polyolefins Co., Ltd. The properties of l-HDPE and h-HDPE are given in Table 3.1

Table 3.1 Physical properties of l-HDPE and h-HDPE*

Grade	Properties	Units	Test result	Test method
l-HDPE	Melt Flow Rate	g/10 min	7.5	ASTM D 1238
	Density	g/cm^3	0.964	ASTM D 1505
	Melting point	°C	133	ASTM D 2117
h-HDPE	Melt Flow Rate	g/10 min	18	ASTM D 1238
	Density	g/cm3	0.962	ASTM D 1505
	Melting point	°C	131	ASTM D 2117

*Data supplied by SCG Polyolefins Co., Ltd

3.1.2 Poplypropylene (PP)

Polypropylene employed in the study was l-PP and h-PP supplied by SCG Polyolefins Co., Ltd. The properties of l-PP and h-PP are given in Table 3.2

Table 3.2 Physical properties of l-PP and h-PP **

Grade	Properties	Units	Test result	Test method
l-PP	Melt Flow Rate	g/10 min	2.6	ASTM D 1238
	Density	g/cm^3	0.910	ASTM D 1505
	Melting point	°C	163	ASTM D 2117
h-PP	Melt Flow Rate	g/10 min	11	ASTM D 1238
	Density	g/cm3	0.910	ASTM D 1505
	Melting point	°C	163	ASTM D 2117

**Data supplied by SCG Polyolefins Co., Ltd

3.1.3 Maleic Anhydride (MAH)

Maleic anhydride (MAH) used for the study was supplied by SIGMA-ALDRICH. The properties of MAH are given in Table 3.3

Table 3.3 Physical properties of maleic anhydride ***

Properties	Units	Test result
Molecular Weight	-	98.06
Vapor pressure	mmHg (20 °C)	0.16
Boiling point	°C	200
Melting point	°C	51-56

***Data supplied by SIGMA-ALDRICH.

3.1.4 Dicumyl Peroxide (DCP)

Dicumyl peroxide (DCP) used for the study was supplied by SIGMA-ALDRICH. The properties of DCP are given in Table 3.4

Table 3.4 Physical properties of dicumyl peroxide ****

Properties	Units	Test result
Molecular Weight	-	270.37
Vapor pressure	mmHg (38 °C)	15.4
Density	g/mL	1.56
Melting point	°C	39-41

****Data supplied by SIGMA-ALDRICH.

3.1.5 Solvent

Toluene AR. grade was supplied by Labscan.

Ethanol AR. grade was supplied by Labscan.

3.1.6 Base Substance

Potassium hydroxide (KOH) was supplied by Carlo Erba.

3.1.7 Acid Solutions

Hydrochoric acid (HCl) 37% wt/v was supplied by J.T. Baker.

3.1.8 Indicator Reagent

Phenopthalein was supplied by Ajex Finechem.

3.2 Equipment

3.2.1 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra of the graft materials are obtained from film samples. These film samples were prepared using a compression-molding machine. Fourier Transform Infrared Spectrophotometer (FT-IR) was used to confirm the successful of grafting, using 64 scans at a resolution of 4 cm^{-1}.

3.2.2 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) measurements were carried out under nitrogen, on samples of 9-15 mg, using a TA Instruments Q-100-instrument. The analysis was carried out in the following manner: (i) Samples were heated from -85 to 210°C at 10°C/min. (ii) Samples were then cooled from 210°C to -85°C at 10°C/min. (iii) After that, samples were immediatelyheated from -85°C to 250°C at 10°C/min

3.2.3 Compression Molding

A Labtech 25 ton compression press machine was used for to prepare samples in a thin film. Pellets were placed in a picture frame mold and the mold preheated at 210°C for 5 minutes in the press without application of pressure. The mold was then compressed under a force of 130 kg/cm^2 for a further 5 minutes after which the mold was cooled 5 minutes to room temperature.

3.2.4 Capillary Rheometer

A CEAST Rheologic 5000 twin-bore capillary rheometer was used to investigate the melt rheology of the grafted materials. The capillary diameter of 1 mm (L/D = 20) and all measurement were made at 190°C.

3.2.6 Wide-angle X-ray Diffraction (WAXS)

The samples were cut from the compression molded plates. All experiments were carried out the 2θ range of 4°- 60° at ambient temperature.

3.2.7 Twin Screw Extruder

The materials were blended in a co-rotating twin screw extruder using a screw speed of 20 rpm. HDPE-g-MAH samples were prepared at the temperature 110 °C to 170 °C. PP-g-MAH samples were prepared at the temperature 130 °C to

170 °C. The strips from the extruder were cut into pellets about 4 mm long after cooling in a water bath.

3.3 Methodology

3.3.1 Preparation of HDPE-g-MAH

HDPE-g-MAH samples were prepared by using a co-rotating twin-screw extruder. The diameter of the screws is 20 mm and the ratio of length to diameter, L/D, is 40. The barrel of extruder is divided into 10 segments and each segment is heated independently. Two different commercial grades of HDPE and varying in MFI were used. The dicumyl peroxide acts as an initiator and varies MAH content and initiator concentration. Rotating rates of the feeder screw was 20 rpm. Under these conditions, the residence time was about 2 min.

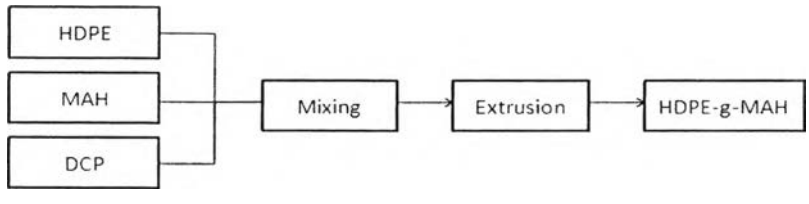

Figure 3.1 Preparation of maleated HDPE.

3.3.2 Preparation of PP-g-MAH

PP-g-MAH samples were prepared by using a co-rotating twin-screw extruder using the same procedure as described above.

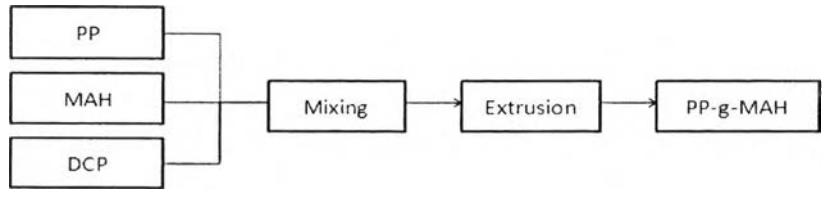

Figure 3.2 Preparation of maleated PP.

3.3.3 Back-titration Method

Grafted materials (5 g) were dissolved in 100 ml toluene. The hot solution was refluxed for 30 min. Then, water (3 ml) was added to hydrolyzed the anhydride groups, after 40 min stirring, the hot mixture was titrated immediately with 0.01N ethanol KOH using three to four drops of phenolphthalein as an indicator, and the pink color was back -titrated to a colorless by the addition of 0.01 N ethanol HCl.

$$G\,(\%\mathrm{wt}) = \frac{(V_0 - V_1)}{2 \times W \times 1000} \times 98 \times 100\%$$

Where, G (wt %) = the grafting degree of maleic anhydride

V_0 (ml) = the KOH volume used in the test with the sample

V_1 (ml) = the HCL volume used in the test with the sample

W = the weight of the sample (g)

Table 3.5 Composition of graft materials (Effect of DCP)

Materials	No.	MR[g]	DCP[phr]	MAH[phr]
l-PP	1	100	0.04	5
	2	100	0.3	5
	3	100	0.6	5
	4	100	2	5
	5	100	5	5
h-PP	1	100	0.04	5
	2	100	0.3	5
	3	100	0.6	5
	4	100	2	5
	5	100	5	5
l-HDPE	1	100	0.08	4
	2	100	0.125	4
	3	100	0.25	4
	4	100	0.5	4
	5	100	1	4
h-HDPE	1	100	0.08	4
	2	100	0.125	4
	3	100	0.25	4
	4	100	0.5	4
	5	100	1	4

Table 3.6 Composition of graft materials (Effect of MAH)

Materials	No.	MR[g]	DCP[phr]	MAH[phr]
l-PP	1	100	0.3	0.04
	2	100	0.3	1
	3	100	0.3	2
	4	100	0.3	6
	5	100	0.3	10
h-PP	1	100	0.3	0.04
	2	100	0.3	1
	3	100	0.3	2
	4	100	0.3	6
	5	100	0.3	10
l-HDPE	1	100	0.125	0.04
	2	100	0.125	1
	3	100	0.125	2
	4	100	0.125	6
	5	100	0.125	10
h-HDPE	1	100	0.125	0.04
	2	100	0.125	1
	3	100	0.125	2
	4	100	0.125	6
	5	100	0.125	10

Table Detail of extrusion processing

Zone/ Conditions	1	2	3	4	5	6	7	8	9	10
Temperature (°C) for PE	110	120	160	160	170	170	170	170	170	160
Temperature (°C) for PP	130	150	165	165	170	170	170	170	170	165
Screw speed (rpm)	20									

Table 3.8 Detail of compression molding

Step	Temperature (°C)	Compressing force (kg/cm^2)	Time (min)
Preheat	210	0	5
Compressing	210	130	3
Cooling	210 to T_{room}	130	5