

CHAPTER III EXPERIMENTAL

3.1 Materials

The HDPE used in this study was a commercial grade of Thai Polyethylene Co., Ltd. (H6240B). The PVC resin (K66) was supplied by Thai Plastic and Chemical Co., Ltd.. Selected characteristics of those polymers are gathered in Table 3.1.

Table 3.1 Selected properties of HDPE and PVC used in this study.

	M_w (g/mol)	M_n (g/mol)	M_w/M_n
H6240B	154,870	12,750	12.15
K66	80,947	70,982	1.14

The functional monomer, MMA, was purchased from Fluka (stabilized with 0.0025 % hydroquinone) and used as received. The free radical grafting of MMA was initiated by dicumyl peroxide (DCP), a commercial grade of Fluka (purity: 97 %). Its half-life time is about 1 hour at 135 °C and 0.2 hour at 150 °C.

3.2 Methodology

3.2.1 Preparation of The Blends

The blending processes were prepared in Brabender Plasti-Corder PL-2100 batch mixer. Temperature and torque were monitored continuously. In all cases rotor speed was 30 rpm.

The electrically heated mixer chamber was preheated to 100 °C and filled with HDPE. After mixing for 5 min, the desired composition of MMA

was added dropwise followed by DCP. The temperature was increased to 160 °C and held there for 5 min. As in all case of MMA grafting, no torque increase was observed. After held for 5 min, PVC was feed into the mixer and mixed for 5 min. Blend composition studied were 90:10, 70:30, and 50:50, HDPE/PVC in term of weight ratio. In order to prevent possible degradation, 2.0 wt % of lead stabilizer was added to the blends.

3.2.2 HDPE Grafted MMA Characterization

3.2.2.1 MMA Grafting Yield Measurement

The amount of the MMA grafted onto HDPE was measured just before the feeding point of the PVC. MMA grafted HDPE samples were taken therefrom and then quenched immediately in liquid nitrogen. After that 0.5 gram grafted HDPE samples were dissolved in 50 ml o-dichlorobenzene and precipitated by addition to a large amount of acetone at room temperature so that residual monomer and possible homopolymer of PMMA were washed out. Next, the purified HDPE samples were dried at 110 °C for 2 hours and then were pressed into thin films. The FTIR spectra of these films were acquired using Bruker FRA 106/S spectrometer at resolution of 2 cm⁻¹ (16 scans per sample). The percent grafting is an approximated value based on the ratio of the carbonyl stretch in HDPE-g-MMA (ν_{CO} , 1730-1740 cm⁻¹) to the internal reference band which is C-H bending in methyl group of HDPE (δ_{CH_3} , 1380 cm⁻¹) in the IR spectra. After deciding upon the condition for the analysis, it is necessary to prepare a calibration curve from a series of samples that bracket the MMA concentration range expected for the experimental. In this study, the calibration curve is a plot of integral ratio of carbonyl stretch band to C-H bending band as a function of percent weight of MMA in standard sample. The standard samples were prepared by melt blending HDPE with poly(methyl methacrylate) (PMMA). The percentage MMA in

standard sample were calculated from the oxygen content which obtained from elemental analysis of oxygen by PE 2400 II CHNS/O Analyzer. Weight percentage of MMA in samples were calculated by follow (Boutevin *et al.*, 1996):

$$\text{Percentage MMA in sample} = [\text{Experimental oxygen content}] \times 100 / 32$$

Figure 3.1 show the calibration curve used for determination of MMA grafting yield in this study.

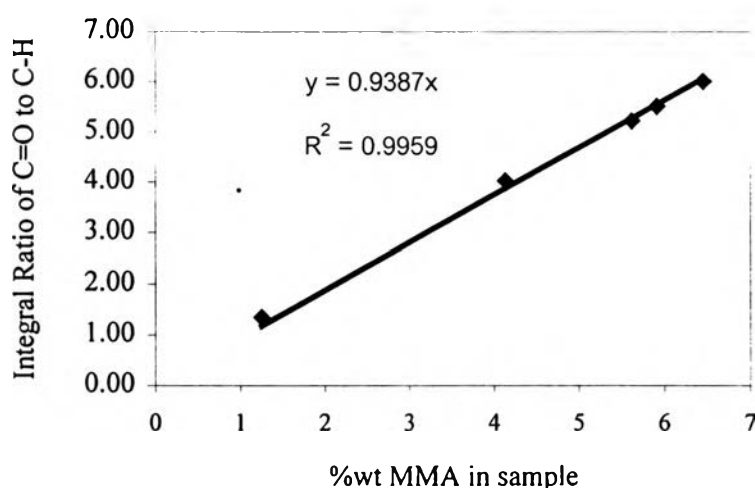


Figure 3.1 Calibration curve for MMA grafting yield measurement.

3.2.2.2 Molecular Weight Characterization

Gel permeation chromatography (GPC) was performed on the sol fractions of MMA grafted HDPE by mean of a Water 150C chromatograph at 140 °C using o-dichlorobenzene as solvent.

3.2.2.3 Melt Flow Index Determination

Melt flow index (MFI) determination was performed on all MMA grafted HDPE pelletized sample according to ASTM 1238-E in the melt indexer (Zwick4105). In most cases, MFI was measured at 190 °C using load

10 kg and 10 minutes extrudate were collected. The results are reported in term of the extrudate weight in gram per 10 minutes.

3.2.2.4 Gel Content Determination

The amount of insoluble portion (gel) was determined from the residual portion of the extracted MMA grafted HDPE samples. The extraction was done by refluxing 1 gram of MMA grafted HDPE in 100 ml o-dichlorobenzene for 24 hour.

3.2.3 HDPE/PVC Blends Characterization

3.2.3.1 Morphological Characterization

To investigate the morphology and phase structure of HDPE/PVC blends, the specimens were fractured under cryogenic conditions using liquid nitrogen. A JEOL JSM 5200 scanning electron microscope (SEM) at 5 kV was used for observation of samples whose surfaces were coated with thin layers of gold.

3.2.3.2 Mechanical Characterization

The specimens for the tensile (ASTM D638) and impact tests (ASTM D256) were prepared by compression molding at 160 °C. After a preheating time 3 min, the specimens were compression molded by applying force of 15 ton for 2 min. The tensile properties were measured by Instron 4206 tensile tester at room temperature, using a crosshead speed of 5 mm/min. The impact strength was done at room temperature with a Zwick impact tester using 2.7 Kg striker.

3.2.3.3 Thermal Characterization

A Netzsch DSC 2000 differential scanning calorimeter was employed to study the glass transition and melting behavior of the samples. The measurements were conducted under nitrogen atmosphere. The sample weight used in DSC cell was kept in the range of 2-10 mg. The samples were then scanned in the apparatus from 50 to 150 °C at heating rate of 10 °C/min. The midpoint of the slope change of the heat capacity plot of the second scan was taken as the glass transition temperature (T_g). The melting point (T_m) was taken as the maximum of the melting endotherm. Crystallinity was calculated for all scans using a theoretical heat of fusion for 100 % crystalline polyethylene of 290 J/g.