

CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Materials

3.1.1 Polypropylene (PP)

General purpose polypropylene homopolymer (Moplen HP400K, 6531), from HMC polymer Co., Ltd. was used. It had melt flow rate of 4 dg/min and density 0.9 g/cm³.

3.1.2 High Density Polyethylene (HDPE)

Injection grade high density polyethylene (Polene, R1760), from Thai Petrochemical Industry Public Co., Ltd. was used. It had melt flow rate of 6 dg/min and density 0.957 g/cm³.

3.1.3 Low Density Polyethylene (LDPE)

Injection grade low density polyethylene (LD 1450J), from Thai Polyethylene Co., Ltd. was used. It had melt flow rate 50 dg/min and density 0.914 g/cm³.

3.1.4 Linear Low Density Polyethylene (LLDPE)

Cast film grade linear Low Density Polyethylene (1221G1), from The Dow Chemical Co., Ltd. was used. It had melt flow rate 2 dg/min and density 0.918 g/cm³.

3.1.5 Reclaimed Tire Rubber (RTR)

Reclaimed tire rubber (RTR) with particle size distribution of 500-600 μm was purchased from Union Commercial Development Co., Ltd. The RTR comprised of 67.57% rubber, 25.35 % carbon black and 7.08% residue.

3.1.6 Crosslinking Agents and compatibilizer

Crosslinking agents and compatibilizer used in this work were obtained from various suppliers as shown in Table 3.1

Table 3.1 Source of materials

Materials	Company
Sulphur (S)	Merck
Tetramethylthiuramdisulfide (TMTD)	Fluka
2-Mercaptobenzothiazole (MBT)	Fluka
Zinc oxide (ZnO)	Merck
Stearic acid	Merck
Maleic anhydride (MA)	Merck
Dicumyl peroxide (DCP)	Fluka
Maleic anhydride grafted polypropylene (MZ203D)	Dupont
Maleic anhydride grafted polyethylene (MB226D)	Dupont

3.2 Instruments

- 3.2.1 Two Roll Mill, LAB TECH (Engine Company)
- 3.2.2 Impact Tester
- 3.2.3 Compression molding machine, LAB TECH
- 3.2.4 Tensile Tester, Universal Testing Machine
- 3.2.5 Thermogravimetric Analyzer, Perkin Elmer Peris Diamond
- 3.2.6 Differential Scanning Calorimeter, Mettler Toledo Star S.W.7.01
- 3.2.7 Scanning Electron Microscope
- 3.2.8 Rotational Rheometer, Rheometric Scientific Model ARES

3.3 Experimental Procedure

3.3.1 Blend preparation

Reclaimed tire rubber (RTR) was well melt mixed with thermoplastic (PP, HDPE, LDPE, LLDPE) at different composition (RTR/thermoplastic 30/70-70/30) on two-roll mill (temperature of the front and back 175°C and 180°C for PP, 110°C and 115°C for LDPE, 135°C and 140°C for HDPE, LLDPE). In all case, thermoplastic was melted first for 2 min before the addition of the RTR. After 10 minutes of mixing, one type of vulcanizing agent (sulphur, MA/DCP, mixed system consisting of sulphur and MA/DCP) and compatibilizer, according to formulations given in Table 3.2, were added and allowed to mix for another 5 minutes and finally sheeted out on the mill. They were then compression molded in an electrically heated hydraulic press for 5 minutes (temperature and pressure 200°C and 10 Kgf/cm² for PP, 150°C and 30.6 Kgf/cm² for LDPE, 190°C and 40.8 Kgf/cm² for LLDPE, HDPE) and subsequently cooled under pressure by water circulation through the platens.

Table 3.2 Recipes of vulcanization and compatibilization system

Ingredients	Amount used (phr)			
	Sulphur	Peroxide	Mixed	Compatibilizer
ZnO	5	-	5	-
Stearic acid	2	-	2	-
MBT	0.75	-	0.75	-
TMTD	1.5	-	1.5	-
Sulphur	3	-	1.5	-
MA	-	2	1	-
DCP	-	0.4	0.2	-
MB226D	-	-	-	10(HDPE), 1(LDPE, LLDPE)
MZ203D	-	-	-	0.3 (PP)

3.3.2 Measuring Notch-Izod Impact Strength

The impact properties were measured according to ASTM D256. Impact specimens had dimensions of 63.5 by 1.27 by 3 mm. The results are reported in Kg-cm/cm notch unit.

3.3.2 Measuring Tensile Strength

Tensile strength measurement was performed on dumbbell specimens at ambient temperature according to ASTM D412 using universal testing machine.

3.3.3 Determination Solvent Swelling

Samples were cut into disc approximately 1 cm diameter and 0.3 cm thickness, then weighed accurately before being immersed into toluene. The samples were kept in a dark place for 7 days at room temperature. Excess liquid on the surface of the sample was removed by blotting with filter paper and weighed quickly.

3.3.4 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was carried out from 50 to 850 °C with a programmed heating rate of 20 °C/min in nitrogen atmosphere with the help of a Perkin Elmer Peris Diamond Thermogravimetric analyser.

3.3.5 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetric studies were carried out using Mettler Toledo Star SW7.01 differential scanning calorimeter. The scans were taken in the temperature range from -80 to 160 °C with a programmed heating rate of 10°C/min. Glass transition temperature, melting temperature and degree of crystallinity were recorded.

3.3.6 Scanning Electron Microscopy (SEM)

SEM photomicrographs of fractured surface were taken in a scanning electron microscope (SEM) in order to investigate the morphology of various blend systems. The fracture surfaced samples were immersed in liquid nitrogen and gold-coated prior to SEM study.

3.3.7 Rheological Studies

Rheological behavior of the blends was studied using Scientific model ARES rheometer, parallel plate and plate rheometer, diameter 25 mm, gap between plate 1 mm., strain 5% and temperature 140°C for HDPE and LLDPE, 115°C for LDPE and 180°C for PP.



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