

CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Materials

3.1.1 Reclaimed tire rubber (RTR)

RTR was purchased from Union Commercial Development Co., Ltd. The RTR was comprised of 63.25% rubber, 22.53% carbon black, 4.66% calcium carbonate and 7.52% residue.

3.1.2 Polypropylene (PP)

PP used in this study was an injection grade (P851JO, melt flow rate of 20 g/ 10 min and density of 0.91 g/cm³). The sample was provided by Siam Cement Co., Ltd.

3.1.3 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 crosslinking agents and compatibilizer

Materials	Company
Sulphur (S)	Merck
Tetramethylthiuram disulfide(TMTD)	Fluka
2-Mercaptobenzothiazole (MBT)	Fluka
Zinc oxide (ZnO)	Merck
Stearic acid	Merck
Propylene octene copolymer (Affinity PL 1881)	Dupont
Maleic anhydride grafted polypropylene (Fusabond P MD 353D)	Dupont

3.2 Instruments

The major instruments used are listed below.

1. Internal mixer (Thermo Haake Rheocord300p)
2. Compression molding machine, LAB TECH
3. Tensile Tester, Universal Testing Machine (Instron Model 55R4502)
4. Instrumented Impact Tester (Radmana Model ITR 2000)
5. Thermogravimetric Analyzer, Perkin Elmer Peris Diamond
6. Nuclear Magnetic Resonance (Advanced 300 MHz Digital NMR)
7. Scanning Electron Microscope (JSM 5410& EDS)
8. Rotational Rheometer, Rheometric Scientific Model ARES (Advanced Rheometric Expansion System)
9. Melt flow indexer, model 10, Davenport (Lloyd Instruments)

3.3 Experimental Procedure

3.3.1 Blend preparation

Reclaimed tire rubber (RTR) was well melt-mixed with PP at different composition (RTR/PP 20/80-80/20) at 180°C in an internal mixer (Thermo Haake Rheocord300p). PP was melted first for 2 minutes before the ZnO was introduced. After 30 seconds of mixing RTR/compatibilizer or virgin rubbers were added into the mixer. After further 5 minutes of mixing, vulcanizing system (sulphur, MBT, TMTD and stearic acid), according to formulations given in Table 3.1, were added and then allowed to mix for 5 more minutes. They were then compression molded in an electrically heated hydraulic press from a sandwich mold with a middle frame made from a 2 mm thick aluminum sheet. The blends prepared by batch processing were placed between the platens, which were preheated to 200°C. The blend was preheated for five minutes and then gradually compressed to a piston pressure of 18 ton for an additional five minutes. Subsequently, the blend was cooled under pressure by water circulation through the platens. The sheet was then removed and die-cut for the appropriate tests.

Table 3.2 Formulation of PP/RTR blends

Blends No.	PP (pbw)	RTR (pbw)	TMTD (phr)	MBT (phr)	Stearic acid (phr)	Sulphur (phr)
1	20	80	1.50	0.75	2	2
2	30	70	1.50	0.75	2	2
3	40	60	1.50	0.75	2	2
4	50	50	1.50	0.75	2	2
5	60	40	1.50	0.75	2	2
6	70	30	1.50	0.75	2	2
7	80	20	1.50	0.75	2	2

Table 3.3 Formulation of PP/RTR blends with additives.

Blends No.	PP (pbw)	RTR (pbw)	POE (phr)	MA-g-PP (phr)	TMTD (phr)	MBT (phr)	Stearic acid (phr)	Sulphur (phr)
1	70	30	3	-	1.50	0.75	2	2
2	70	30	5	-	1.50	0.75	2	2
3	70	30	7	-	1.50	0.75	2	2
4	70	30	9	-	1.50	0.75	2	2
5	70	30	-	3	1.50	0.75	2	2
6	70	30	-	5	1.50	0.75	2	2
7	-	-	-	7	1.50	0.75	2	2
8	-	-	-	9	1.50	0.75	2	2

Table 3.4 Formulation of PP/RTR blends with virgin rubber.

Blends No.	PP (pbw)	RTR (pbw)	NR (pbw)	EPDM (pbw)	TMTD (phr)	MBT (phr)	Stearic acid (phr)	Sulphur (phr)
1	70	30	10	-	1.50	0.75	2	2
2	70	30	20	-	1.50	0.75	2	2
3	70	30	30	-	1.50	0.75	2	2
4	70	30	-	10	1.50	0.75	2	2
5	70	30	-	20	1.50	0.75	2	2
6	70	30	-	30	1.50	0.75	2	2

For RTR swelling test .To prepares vulcanization and unvulcanized by with and without sulphur crosslinking agent and compression molded.

3.3.2 Measuring Notch-Izod Impact Strength

The impact properties were measured according to ASTM D256 Impact specimens had dimensions of 63.5 x 1.27 x 3 mm. The results are reported in J/m notch unit.

3.3.3 Measuring Tensile Strength

Tensile strength measurement was performed on dumbbell shape specimens at ambient temperature according to ASTM D412 using an universal testing machine Tensile Tester, Universal Testing Machine (Instron Model 55R4502).

3.3.4 Determination of Solvent Swelling

Determination of the swelling percentage of blends was carried out according to ASTM D 471. Samples dimension 30 x 5 x 2 mm were weighed using an electric

balance and this was considered to be the original weight (W_1). The test 3 samples were immersed in ASTM toluene at room temperature for 48 hours. The samples were then removed from toluene, wiped with tissue paper to remove excess toluene, and weighed (W_2). The swelling percentage of the blends was then calculated as follow:

$$\text{Swelling percentage} = \frac{W_2 - W_1}{W_1} \times 100\% \quad \dots \quad \text{Equation 3.1}$$

3.3.5 Nuclear magnetic resonance (NMR)

The NMR experiments were performed at solid state carbon nuclear magnetic resonance spectroscopy (^{13}C -NMR) frequency of 75 MHz on AVANCE 300 MHz Digital NMR. All solid NMR experiments were done at CP Mass probe and were performed using gated high decoupling. A zirconium oxide rotor of 7 mm diameter was used to acquire the NMR spectra at a spinning rate of 5.0 kHz.

3.3.6 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was carried out under the range of temperature from 40 to 850°C with a programmed heating rate of 20°C/min in nitrogen atmosphere by using a Mettler Toledo Star Thermogravimetric analyzer.

3.3.7 Scanning Electron Microscopy (SEM)

SEM photomicrographs of fracture surface were taken in a scanning electron microscope (SEM) in order to investigate the morphology of various blend systems. The samples were fractured in liquid nitrogen, stained with osmium tetroxide (OsO_4) and coated with carbon for the study.

3.3.8 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 0.5 mm, strain 5% and temperature 180°C.

3.3.9 Measuring melt flow index (MFI)

Measurement of MFI (g/10 min) was carried out according to ASTM D 1238, utilizing a melt flow indexer, model 10, Davenport (Lloyd Instruments). The blend sample of 3-4 g was heated at 230 ° C extrusion plastometer and was compressed with 2.16 kg dead load. The sample weight flow out was recorded about 10 minutes.