

CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Materials

3.1.1 Polypropylene (PP)

Injection grade homopolymer polypropylene (PRO FAX 6531), from HMC polymer Co., Ltd. was used. It had a melt flow rate of 4 dg/min and density of 0.9 g/cm³.

3.1.2 Recycled Tire-Rubber

There are two types of recycled tire-rubber, ground rubber tire (GRT) and reclaimed tire- rubber (RTR), which most of the metal and polyester cord had been separated. Both types were from Union Commercial Development Co., Ltd. In case of GRT, three different mesh sizes were used (Table 3.1).

Table 3.1 The different mesh sizes of GRT

Code No.	Particle Size	
	mesh	mm.
GRT 8	8	2.38
GRT 16	16	1.19
GRT 40	40	0.42

3.1.3 Antioxidant

Bis(2,4-di-t-butyl phenyl) Pentaerythritol Diphosphite Antioxidant (Ultranox 626), GE Specialty Chemicals, supplied by Nagase (Thailand) Co., Ltd. were used.

3.1.4 Crosslinking Agents

In this study, either one of two crosslinking systems, sulfur crosslinking agent and MA/DCP was used (as shown in Table 3.2)

Table 3.2 The components in crosslinking agents

Crosslinking agent	Component	Supplier
Sulfur Crosslinking	Sulfur (S)	MAY&BAKED LTD. DAGENHAM ENGLAN
	Tetramethyl thiuramdisulfide (TMTD)	FLUKA
	2-Mercaptobenzothiazole (MBT)	FLUKA
	Zinc oxide (ZnO)	BDH CHEMICALS LTD. ENGLAND
	Stearic acid	MAY&BAKED LTD. DAGENHAM ENGLAND
MA/DCP	Maleic anhydride (MA)	MERCK
	Dicumyl peroxide (DCP)	FLUKA

3.2 Instruments

The major instruments used are listed below.

1. Two-Roll Mill , LAB TECH (Engine Company)
2. HAAKE Rheomex CTW 100P Twin Screw Extruder (Germany)
3. TOSHIBA IS 100G Injection Molding Machine (Tokyo, Japan)
4. KEYNESS D 7053 Automatic Melt Flow Indexer (Morgantown, PA)
5. Pendulum Impact Tester
6. JEOL JSM 5800LV Scanning Electron Microscope (Japan)
7. PERKIN ELMER DSC7 Differential Scanning Calorimeter (Connecticut,USA)
8. PERKIN ELMER TGA7 Thermogravimetric Analyzer (Connecticut, USA)
9. BOSCO Crushing
10. Oertling NA 244 Weighter
11. Pelletizer (Thai Hydraulic machinery Co., Ltd.)

3.3 Experimental Procedure

3.3.1 Recycled Tire-Rubber Compositions

The determination of the main components composition of reclaimed tire-rubber (RTR) and ground rubber tire (GRT) was carried out using thermogravimetric analyzer (TGA), heated from 50°C to 850°C at a scanning rate of 20°C/min under oxygen atmosphere. The glass-transition temperature of RTR was investigated by differential scanning calorimeter (DSC), heated from -100°C to 50°C, scanning rate of 20°C/min under helium atmosphere, which confirmed the miscibility of each type of rubber in RTR.

3.3.2 Composites Preparation

Homopolypropylene(PP) was well melt-mixed with either reclaimed tire-rubber(RTR) or ground rubber tire (GRT) at different composition in the presence of either sulfur crosslinking agent or MA/DCP system according to formulations given in Table 3.3-3.5.

Each mixture was firstly melt-mixed in a two-roll mill. The temperature of the front and back roll were set at 170°C and 165°C respectively. The composites were normally cut diagonally from time to time and folded over several times during mixing. After 10 minutes of mixing, one type of vulcanizing agents and antioxidant (ultranox 626) were added and allowed it mix for another 5 minutes before taking off the roller. The blend was chopped and feeded into a counter-rotating twin screw extruder and then palletized. The extruder temperature profile was set at 190/195/200 and 205°C for die heating zone and the screw rotating speed was set at 80 rpm. This pallet sample were molded to form dumbbell-shaped tensile specimens and impact specimens by injection molding machine at 215/220/225/220°C (from hopper to nozzle).

Table 3.3 Formulation of PP/RTR blends using sulfur crosslinking agent.

Composite No.	PP (pbw)	RTR (pbw)	Component					
			TMTD (phr)	MBT (phr)	ZnO (phr)	Stearic Acid (phr)	Sulfur (phr)	AO (pbw)
S1	100	-	-	-	-	-	-	1.0
S2	80	20	-	-	-	-	-	1.0
S3	80	20	1.5	0.75	5.0	2.0	3.0	1.0
S4	75	25	-	-	-	-	-	1.0
S5	75	25	1.5	0.75	5.0	2.0	3.0	1.0
S6	70	30	-	-	-	-	-	1.0
S7	70	30	1.5	0.75	5.0	2.0	3.0	1.0
S8	65	35	-	-	-	-	-	1.0
S9	65	35	1.5	0.75	5.0	2.0	3.0	1.0
S10	60	40	-	-	-	-	-	1.0
S11	60	40	1.5	0.75	5.0	2.0	3.0	1.0

Table 3.4 Formulation of PP/RTR blends using MA/DCP.

Composite No.	PP (pbw)	RTR (pbw)	MA (pbw)	DCP (pbw)	AO (pbw)
S12	70	30	-	-	1.0
S13	70	30	2	-	1.0
S14	70	30	-	0.4	1.0
S15	70	30	2	0.4	1.0

Table 3.5 Formulation of PP/GRT blends using sulfur crosslinking agent.

Composite No.	PP (pbw)	Ground Rubber Tire (pbw)			Crosslinking Agent					
		GRT 40	GRT 16	GRT 8	TMTD (phr)	MBT (phr)	ZnO (phr)	Stearic acid (phr)	Sul-fur (phr)	AO (pbw)
S16	70	30	-	-	-	-	-	-	-	1.0
S17	70	30	-	-	1.5	0.75	5.0	2.0	3.0	1.0
S18	70	-	30	-	-	-	-	-	-	1.0
S19	70	-	30	-	1.5	0.75	5.0	2.0	3.0	1.0
S20	70	-	-	30	-	-	-	-	-	1.0
S21	70	-	-	30	1.5	0.75	5.0	2.0	3.0	1.0

3.4 Measuring Notch–Izod Impact Strength

The impact specimens of the blends from injection molding machine with dimensions of 6.35 by 1.27 by 0.64 cm were notched with radius of 0.0254 cm. by using notch cutter. Notch-Izod impact strength of the sample was measured following the procedures described in ASTM D 256, which use 2.7 Joules pendulum swing and performed at room temperature. The energy for breaking was recorded and then converted to impact strength by dividing it with the area of the specimen.

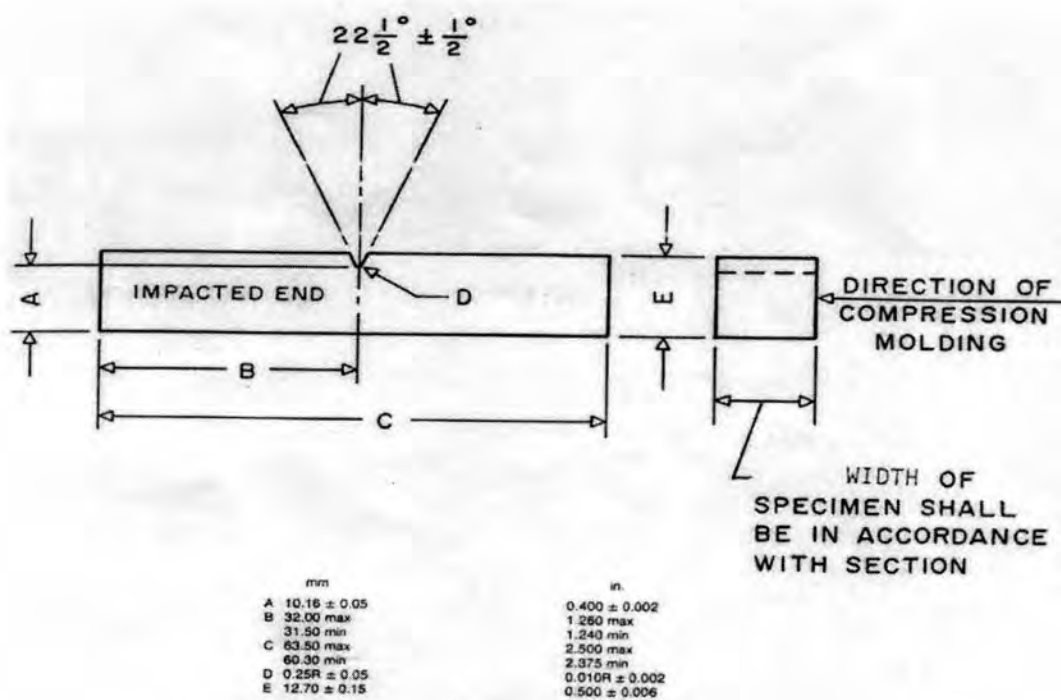


Figure 3.1 Dimensions of Izod-Type Test Specimen [52]

3.5 Measuring Melt Flow Index (MFI)

Measurement of MFI (g /10 min.) was carried out according to ASTM D 1238, utilizing a KEYNESS 7053 melt flow indexer. The injection molded sample of 3-4 g was heated at 230°C in extrusion plastometer and were compressed with 2.16 kg dead load. The sample weight flew out was recorded about 10 minutes.

3.6 Measuring Thermal Properties

Thermal properties were measured by differential scanning calorimeter (DSC). A sample of injection-molded specimens of 10-15 mg. was first heated at room temperature and gradually rising to 185°C at the heating rate of 20°C/min. The onset temperature, melting temperature and heat of fusion (H_f) of the composites were recorded. The area of the melting peak, endothermic, was directly equal to the heat of fusion.

3.7 Examination of Surface Morphology

The untested dumbbell-shaped tensile specimens from injection mold were immersed in liquid nitrogen, and then fractured. The surfaces of this fractured sample was stained with osmium tetroxide (OsO_4), 17 hours at room temperature and stubs with double-sided adhesive tape and sputter coated with gold.

The morphology of the blending was examined by the SEM at 15 kV accelerating voltage, and detected the electrons with backscattered electron emission detector, which produced the higher in image contrast.