CHAPTER III

EXPERIMENTAL

3.1 Materials

Materials used in this research are obtained from various sources and are shown in Table 3.1. Their physical and chemical properties are shown in Appendix I.

Table 3.1 Materials and sources of supplier used in the PP/EPDM composition

Materials	Trade name	Source of Suppliers
Polypropylene	2500 TC	Thai Petrochemical Industry (Public) Ltd.
EPDM	JSR EP35	Japan Synthetic Rubber Co.,Ltd.
Talc	Talcum No.35	San and Soil (Bangkok) Co.,Ltd.
Clay	Clay FK89	San and Soil (Bangkok) Co.,Ltd.
Carbon black	Printex G	JJ-Degussa (T) Ltd.
Glass fiber	CS 3PE-330	Nitto Boseki Co.,Ltd.
HDPE	V1160	Thai Petrochemical Industry (Public) Ltd.
Antioxidant	Irganox 1076	Ciba-Guigy Ltd.
Coupling agent	Siquest A-174	OSI Specialty (Thailand) Ltd.

3.2 Apparatus

1. Two-roll mills: K.Y.S. Co., Ltd. Model MT2-2

2. Crushing machine: Toyo Seiki Co., Ltd. Model 1514
3. Compression molding machine: Shinto Metal Industries Limited Type SFA-50
4. Auto melt indexer: Tester Sangyo Co., Ltd. Model TP-406
5. Impact strength testing: Toyoseiki Co., Ltd. Model 612
6. Flexural strength testing: Instron Corporation Co., Ltd. Model 4302
7. Hardness testing: Instrument&MFG Co., Ltd. Model 2000 Type D
8. Differential scanning colorimeter: Perkin Elmer Co., Ltd. Model DSC7
9. Scanning electron microscopy: JEOL Type JSM-6400
10. X-ray fluorescence spectrometry: PHILIPS Type PW 2400

3.3 Experimental procedure

Various composites were prepared by varying the amount of PP, EPDM and reinforced additives such as talc, clays, carbon black, glass fiber and HDPE (Table 3.2). All components of the preparations were part by weight of total amount of PP and EPDM. Antioxidant at 0.2 wt% was added into all compositions. Finally, mechanical properties MFI, NI, FS and Hardness of each composition were measured and compared with those from two commercial resins A and B.

3.3.1 Mixing procedure

The compositions were prepared by blending all of the components on a two-roll mills at 165°C (21). The PP was first introduced to preheat for 1-2 minute and followed by EPDM, antioxidant and reinforces additives, respectively. A batch was mixed for at least 10 minutes after blending. Distance between the rolls (the roll nip) was adjusted to facilitate mixing.

3.3.2 Molding procedure

The sample was heated in an oven at 80°C for 1 hour before molding. In the molding process, the temperature of 220°C and pressure of 40 kgf/cm^2 were employed for 8-10 minutes. The cooling rate was 1.5°C/1 min. Then test pieces were cut out of these plates for mechanical measurement.

Table 3.2 Formulation of PP/EPDM blends

Sample No.	Ingredients (wt%)								
	PP	EPDM	talc	clay	cb	gf	HDPE	Coupling agent	Antioxidant
1	90	10	10	-	-	-	-	-	0.2
2	85	15	10	-	-	-	-	-	0.2
3	80	20	10	-	-	-	-	-	0.2
4	75	25	10	-	-	-	-	-	0.2
5	70	30	10	-	-	-	-	-	0.2
6	65	35	10	-	-	-	-	-	0.2
7	60	40	10	-	-	-	-	-	0.2
8	80	20	15	-	-	-	-	-	0.2
9	80	20	20	-	-	-	-	-	0.2
10	80	20	25	-	-	-	-	-	0.2
11	80	20	10	5	-	-	-	-	0.2
12	80	20	10	10	-	-	-	-	0.2
13	80	20	10	15	-	-	-	-	0.2
14	80	20	10	-	5	-	-	-	0.2
15	80	20	10	-	10	-	-	-	0.2
16	80	20	10	-	15	-	-	-	0.2
17	85	15	15	-	15	-	-	-	0.2
18	90	10	15	-	15	-	-	-	0.2
19	90	10	20	-	15	-	-	-	0.2
20	90	10	25	-	15	-	-	-	0.2
21	90	10	30	-	15	-	-	-	0.2
22	90	10	15	-	15	5	-	0.1	0.2
23	90	10	15	-	15	10	-	0.2	0.2
24	90	10	15	-	15	15	-	0.3	0.2
25	90	10	30	-	15	-	5	-	0.2
26	90	10	25	-	15	-	10	-	0.2
27	90	10	30	-	15	-	10	-	0.2

Note: cb = carbon black

gf = glass fiber

3.3.3 Effect of EPDM level

Various amount of EPDM at 10, 15, 20, 25, 30, 35 and 40 wt% of the resin mix were mixed with PP and 10 wt% talc. Mechanical properties of each composition as well as commercial resins A and B were measured.

3.3.4 Effect of reinforced additive loading

The selected formulation from 3.3.3 was mixed with reinforced additives such as talc, clay and carbon black at 5, 10 and 15 wt%. Then mechanical properties of each composition were measured and compared with those of commercial resins A and B.

3.3.5 Effect of PP level

The high PP level at 85 and 90 wt% were mixed with the selected type and loading of reinforced additives from 3.3.4. Then mechanical properties of each composition were measured and compared with those of commercial resins A and B.

3.3.6 Effect of talc loading

Various amount of talc at 20, 25, and 30 wt% were mixed with the selected composition from 3.3.5. Then mechanical properties of each composition were measured and compared with those of commercial resins A and B.

3.3.7 Effect of glass fiber

Various amount of glass fiber at 5, 10, and 15 wt% were mixed with the selected composition from 3.3.5. Silane coupling agent at the amount of 2 wt% of glass fiber was used to provide bonding between glass fiber and resin (20). Then mechanical properties of each composition were measured and compared with those of commercial resins A and B.

3.3.8 Effect of HDPE

Various amount of HDPE fiber at 5 and 10 wt% were mixed with the composition from 3.3.6. Then mechanical properties of each composition were measured and compared with those of commercial resins A and B.

Each composition was prepared twice. The mechanical properties, MFI, NI, FS and hardness, of the specimens were obtained three times for each sample, as shown in Appendix I (Table 1, 2 and 3).

3.3.9 Mechanical measurement

The mechanical properties, MFI, NI, FS and Hardness were obtained by using standard size and shape under standard test methods of the American Society for Testing and Materials (ASTM) which subjected to the following measurement

Melt flow index: testing method of ASTM D1238

Impact strength: testing method of ASTM D256

Flexural strength: testing method of ASTM D790

Hardness: testing method of ASTM D2240

3.3.10 X-ray fluorescence spectrometry

The sample approximately 5 grams was mixed with 10 wt% Boric acid and compressed in the aluminum cell with 5 tons pressure. Then the specimen was put in a sample cup and irradiated with high-energy primary X-ray photon. The crystal used was phosphorus (P) and lithium fluoride (LiF).

3.3.11 Differential scanning calorimeter

The 4.0 to 5.0 mg range of sample was put on aluminum pan with tweezers and then punched a cover to provide atmosphere before testing. The Nitrogen was used as purge gas to control atmosphere in the sample cells. The temperature range of analysis was run from 50 to 200°C at heating rate of 10°C min^{-1}.