CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

In this study, PP of extrusion grade (Polene 1102H) was used as polymer the matrix. The physical properties of the PP are shown in Table 3.1.

Table 3.1 Physical properties of polypropylene

Property	Method	Value
MFI (g/10min)	DIN 53735	1.8
Tensile Strength at Yield (N/mm^2)	DIN 53455	35
Modulus of Elasticity (N/mm^2)	DIN 53457	1400
Impact Strength (kJ/m^2) 0 °C -20 °C	DIN 53453	 70 13
Notched Impact Strength (kJ/m^2) 0 °C -20 °C	DIN 53453	 6 2
HDT (°C)	DIN 53461	95

Remark: The values represented on the above are typical laboratory averages [Ref : Issue No.6699 Rev.No.01(TPI Co.,Ltd.)]

Two different grades of carbon black, Printex 30 and Printex 300 (Printex is a registered trademark of JJ-Degussa (Thailand) Co., Ltd.) were supplied from Degussa Co., Ltd. Some morphological characteristics of the aggregates for these two commercial blacks are summarized in Table 3.2.

Table 3.2 Morphological characteristics of carbon black aggregates

Type	pH value	DBPA (ml/100g)	Tapped density (g/l)	Particle size (nm)	BET surface area (m^2/g)
Printex 30	10.0	106	240	27	80
Printex 300	9.5	65	360	27	80

Note : Data from JJ-Degussa (Thailand) Co., Ltd.

Based on the measured values of DBPA and perimeter fractal dimension, Printex 30 can be classified as a high structure carbon back, whereas Printex 300 can be characterized as a low structure carbon black (Chapter 2.2.1).

PDMS [poly (dimethyl siloxane)] of kinematic viscosity 60,000 cS was used as the matrix fluid for measuring the density of the agglomerate by using a pycnometric technique [Tovmasyan et al.(1983)].

3.2 Experimental Procedure

3.2.1 Agglomerate Preparation

Agglomerates were prepared from six experimental samples of fluffy carbon black (0%H, 20%H, 40%H, 60%H, 80%H, and 100%H). The blending of carbon black formula used in this investigation are summarized in Table 3.3. The powder was dried at 120 °C in the oven to prevent moisture for 1 hr. Agglomerates of the powder were prepared by rolling in glass bottle (50rpm) for 120 minutes, followed by sieving (to 425 μm) to obtain the same agglomerate size.

Table 3.3 Blending of carbon black formulation

Formulation Carbon Black	Blending (%)	
	Low Structure Grade	High Structure Grade
0%H*	100	0
20%H	80	20
40%H	60	40
60%H	40	60
80%H	20	80
100%H	0	100

* : H : High structure carbon black

3.2.2 Agglomerate Density Measurement

The porosity of the prepared agglomerates measured through pycnometry was consistent with the values calculated on the basis of the weight of powder. The density of the individual agglomerates was measured

using a pycnometric technique, assuming that the porosity of the prepared agglomerate was unaffected. The method involves the use of a pycnometer of known volume, then weighed containing the agglomerate carbon black to be evaluated. Finally the pycnometer (containing agglomerate carbon black) is filled with a PDMS of known density and reweighed. The weight and therefore the of volume of PDMS can be found by difference.

Agglomerate density is calculated from the following equation

$$\rho_{aggl} = [W_{agg}/(V_{pyc}-(W_{PDMS}/\rho_{PDMS})]$$

Where :

W_{agg} is the weight of agglomerates in pycnometer

V_{pyc} is the volume of pycnometer = 0.4146 cm^3

ρ_{PDMS} is the density of PDMS = 1.441 g/cm^3

W_{PDMS} is the total weight of PDMS filled in pycnometer

3.2.3 Dispersive Mixing Process

Mixing was studied using a Brabender Plasti-Corder, PL-2100, equipped with a cam type mixer-measuring head. The mixer-measuring head consists of an intra-connected, figure-eight-shaped chamber in which two sigmoid, counter-rotating blades turn. The test sample is confined in the chamber and worked between the blades and the chamber walls. Samples were taken at different mixing times. A rotor speed of 40 rpm and an oil bath temperature of 170°C were used. The actual volume of the mixer-measuring head was 55 cm^3. All experiments were run using a fill factor of 0.80. The polymer was charged to the mixer-measuring head and the torque required to rotate the blades was transmitted from the dynamometer housing to the computer to correct the torque data as a function of time. The polymer

temperature was measured with a thermocouple at the base of the mixer-measuring head. No gas was injected into the mixing chamber.

Polypropylene was first masticated for 2 minutes inside the chamber followed by the addition of carbon black. The torque and polymer melt temperature were recorded as a function of time. Power torque curves were plotted continuously as the mixing proceeded in the Brabender. All experiments were run on Heat and Shear Stability mode.

In order to observe the agglomerate size and distribution, the mixture of carbon black and polypropylene was collected from the Brabender mixing measuring-head every minute for all blending conditions. For each condition, the amount of carbon black loading was varied in the percentage of 5, 10, 15, 20, 25, and 30 to the amount of polymer melt, polypropylene. Total sample weight was 28 g per batch. All experiments were repeated 5 times to obtain average data.

3.2.4 Agglomerate Size Analysis

The size analysis was performed by observation with a Scanning Electron Microscope (SEM), model JSM-35CF. The samples were prepared by dipping into liquid nitrogen 30-60 second, then breaking and coating surface with gold for 300 Å. Semafore program was then applied to analyze the size agglomerate and size distribution of the dispersed carbon black in PP.

3.2.5 Dynamic Mechanical Analysis

The dynamic mechanical test was performed on a Rheometric Sceientific RHIOS instrument using the mode of "dynamic strain sweep default test" at ambient temperature. The effect of carbon black on the dynamic mechanical properties of polypropylene compounds has been extensively studied according to Medalia (1978). Samples at different mixing

times were prepared by compression molding the carbon black/PP compounds at 170 °C to obtain flat sheets. Specimens measuring 25 mm diameter and 2 mm thickness were obtained.