CHAPTER II
EXPERIMENT DETAILS

2.1 Materials

2.1.1 High Density Polyethylene (HDPE)

The High Density Polyethylene (HPDE) used was H5690S Monofilament Yarn & Sheet grade from Siam Chemical Trading Co., Ltd. with a reported molecular weight of 107,236 g/mole, a density of 0.956 g/cm^3 and a melt flow index (190°C/2.16 Kg) of 0.9 g/10 min.

2.1.2 Polypropylene (PP)

The Polypropylene (PP) used were P340J Injection Moulding grade, and P400S Monofilament Yarn & Sheet grade from Siam Chemical Trading Co., Ltd. with a reported molecular weight of 174,380 and 176,644 g/mole and melt flow indices (230°C/2.16 Kg) of 1.8 and 3.5 g/10 min respectively.

2.2 Materials Preparation

2.2.1 Varying Composition of HDPE/PP (P340J) Blends

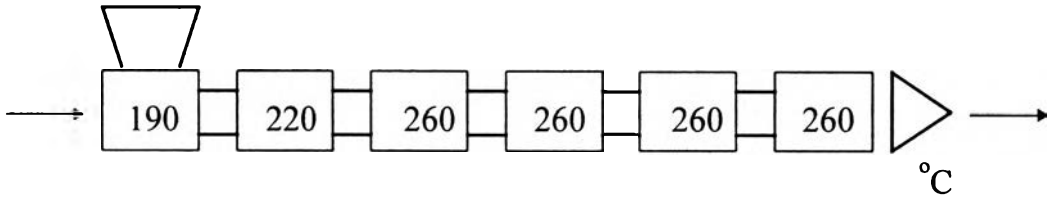
Virgin HDPE was mixed with virgin PP (P340J) at various ratios, namely 0%, 20%, 30%, 40%, 50%, 60%, 70%, 80% and 100% (by weight) by the COLLIN co-rotating twin screw kneader ZK-25 (25mm x 30D).

2.2.2 Varying Melt Flow Index of Two Grades of PP with a fixed HDPE

Virgin HDPE (H5690S) was mixed with two grades of PP, P340J and P400S grades: in 30% (by weight) PP by the COLLIN co-rotating twin screw kneader ZK-25 (25mm x 30D).

All processing passes were carried out with the following conditions based on recommendations from the supplier ;

zone1 zone2 zone3 zone4 zone5 zone6



Screw speed = 50 rpm.

Feed rate = 40 g/min.

The extrudate was cooled in the water (~25 °C) and cut into pellets form by a Planetrol 07502 pelletizer.

2.3 Instruments

2.3.1 Capillary Rheometer

The capillary used was an Instron model 3213 rheometer with a 25 kN load cell, operated in a constant piston speed with a resolution of 0.1 mm/min. The temperature was always set at 180 °C, with a system accuracy of 2.6 °C, for all melt blends studied. The capillary was tapered type with a length of 2.5507 mm and a diameter of 0.7645 mm, giving a length to diameter ratio of 33.4. The diameter of barrel was 9.525 mm.

2.3.2 Rheometer

The pellet samples were used to measure the storage and the loss modulus. The tests were done on Rhometer model RES with a parallel plate, operated in the dynamic frequency sweep temperature default mode with 5% strain and the frequency range of 0.1-100 rad/s. The diameter of parallel plate was 50 mm and the working temperature was varied from 120 °C to 180 °C. The nominal gap size was 1.9 mm.

2.3.3 Stereomicroscope

The Stereomicroscope used was Olympus model SZ4045TR with a magnification range of 0.67 to 6.0 and a 35 mm camera was used for photographic records.

2.3.4 Optical Microscope

The micrographs of extrudate fractures were obtained from Olympus Optical Microscope model BX60, with a magnification range of 500 to 2500 times. The magnification of eyepiece is 10 times and the objective lens are 50-250 times.

2.3.5 Melt Flow Index Meter

Melt Flow Index (MFI) of all samples were determined following ASTM D 1238 on a Zwick 405 Extrusion Plastomer with a piston load of 2.16 kg at 190°C and 230°C respectively. The "die" diameter was 1.18 mm and the die length was 8.00 mm. The melt flow index was calculated as follows :

$$MFI = (\text{weight of sample/cutting time}) \times 600 . \qquad (2.1)$$

2.3.6 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) was carried out on NETZCH (model TASC 414/3). Samples of 8-12 mg. were prepared in aluminum sample pans. The temperature was programmed at a heating rate of 10 °C/min from 30 °C to 300 °C. The chamber was purged with dry nitrogen at a flow rate 25 ml/min. The melting point (T_m), heat of fusion (ΔH_f) were determined from the thermogram by using the DSC standard data analysis V.4.0 (DSC-4.0) software. The corresponding degree of crystallinity of the samples were calculated from the H_f by using the following equation :

$$\text{Percentage crystallinity} = (H_f / H_{fc}) \times 100 \ , \qquad (2.2)$$

where H_f is the heat of fusion of sample from thermogram and H_{fc} is the theoretical heat of fusion of 100 percent crystallinity of the same polymer. H_{fc} of HDPE and PP (Brandrup and Immergut, 1989) are 277.1 and 209 J/g respectively.

2.3.7 Density Measurement

The density of all samples was measured by the density gradient column technique, according to ASTM 1505-85. The density was measured by the flotation level after dropping a polymer sample into the density gradient column at the temperature of 23°C $\pm$ 0.2. The procedure for the preparation of a linear gradient column was by mixing sodium acetate and methanol in various proportionals.

2.4 Characterization

2.4.1 Melt Flow Index Meter

Our procedure followed ASTM D1238. The melt flow indices are shown below in Table 2.4.1.

Table 2.4.1 Melt flow index

Materials	**MFI (g/10min) 190^{0}C/2.16 kg**	**MFI (g/10min) 230^{0}C/2.16 kg**
Pure HDPE	0.9	-
Pure PP (P340J)	-	1.8
Pure PP (P400S)	-	3.5
HDPE/PP (P340J) : 100/0	0.804	1.297
HDPE/PP (P340J) : 80/20	0.609	1.119
HDPE/PP (P340J) : 70/30	0.538	1.008
HDPE/PP (P340J) : 60/40	0.547	1.042
HDPE/PP (P340J) : 50/50	0.542	1.138
HDPE/PP (P340J) : 40/60	0.630	1.312
HDPE/PP (P340J) : 30/70	0.619	1.355
HDPE/PP (P340J) : 20/80	0.662	1.552
HDPE/PP (P340J) : 0/100	0.866	2.132
HDPE/PP (P400S) : 70/30	0.997	1.801

2.4.2 Differential Scanning Calorimetry (DSC)

The melting temperature and the percentage of crystallinity were obtained from DSC. Both properties are shown below in Table 2.4.2.

Table 2.4.2 Melting temperature and percentage of crystallinity

Materials	$T_{m\text{-}HDPE}$ (^{0}C)	$T_{m\text{-}PP}$ (^{0}C)	%Crys.$_{HPDE}$	%Crys.$_{PP}$
Pure HDPE	132	-	-	-
Pure PP (P340J)	-	162	-	-
Pure PP (P400S)	-	170	-	-
HDPE/PP (P340J) : 100/0	135.8	164.7	54.459	-
HDPE/PP (P340J) : 80/20	135.8	164.7	48.130	5.239
HDPE/PP (P340J) : 70/30	134.8	167.2	41.647	8.688
HDPE/PP (P340J) : 60/40	134	168	35.710	11.689
HDPE/PP (P340J) : 50/50	133.3	168.1	30.316	15.296
HDPE/PP (P340J) : 40/60	132.8	168.2	22.686	18.355
HDPE/PP (P340J) : 30/70	132.5	169.3	16.593	21.564
HDPE/PP (P340J) : 20/80	131.3	169.6	10.770	23.420
HDPE/PP (P340J) : 0/100	-	169.1	-	33.743
HDPE/PP (P400S) : 70/30	135.4	165.8	36.812	10.116

Note:

$T_{m\text{-}HDPE}$: Melting Temperature in HDPE Fraction.

$T_{m\text{-}PP}$: Melting Temperature in PP Fraction.

$\%Crys_{HDPE}$: Percentage of Crystallinity in HDPE Fraction.

$\%Crys_{PP}$: Percentage of Crystallinity in PP Fraction.

2.4.3 Density Measurement

ASTM 1505-85 procedure was followed to obtain the density by the density gradient column method. The density values are shown below in Table 2.4.3.

Table 2.4.3 Density

Materials	Density (g/cm^3)
Pure HDPE	0.956
Pure PP (P340J)	-
Pure PP (P400S)	-
HDPE/PP (P340J) : 100/0	0.951
HDPE/PP (P340J) : 80/20	0.940
HDPE/PP (P340J) : 70/30	0.935
HDPE/PP (P340J) : 60/40	0.930
HDPE/PP (P340J) : 50/50	0.926
HDPE/PP (P340J) : 40/60	0.92
HDPE/PP (P340J) : 30/70	0.915
HDPE/PP (P340J) : 20/80	0.910
HDPE/PP (P340J) : 0/100	< 0.910
HDPE/PP (P400S) : 70/30	0.938

2.4.4 Parallel Plate Rheometer

The instrument measures the value of storage modulus (G_g) as shown in Table 2.4.4.

Table 2.4.4 Glassy storage modulus

Materials	**G_g (dyne/cm^2)**
Pure HDPE	-
Pure PP (P340J)	-
Pure PP (P400S)	-
HDPE/PP (P340J) : 100/0	25,100,000
HDPE/PP (P340J) : 80/20	35,710,000
HDPE/PP (P340J) : 70/30	24,230,000
HDPE/PP (P340J) : 60/40	33,350,000
HDPE/PP (P340J) : 50/50	14,570,000
HDPE/PP (P340J) : 40/60	25,220,000
HDPE/PP (P340J) : 30/70	26,640,000
HDPE/PP (P340J) : 20/80	35,380,000
HDPE/PP (P340J) : 0/100	21,560,000
HDPE/PP (P400S) : 70/30	-

Note:

G_g = Storage Modulus at the plateau region or glassy zone.