CHAPTER III

EXPERIMENTAL

This research comprises two parts of work: (1) Study scope and limitation of Simplex equation in predicting mechanical properties of polymer blends, and (2) Preparation and mechanical properties test of polypropylene blend with high-density polyethylene with four different molecular weights in order to study how the interaction parameter(β_{12}) in the Simplex equation depends on molecular weight of HDPE in blends.

3.1 Scope and limitation in using Simplex equation

Mechanical properties of polymer blends as a function of composition were collected from journals and Simplex equation as shown in equation (36) was used to calculate mechanical properties of polymer blends collected. Mechanical properties predicted by Simplex equation and experimental data collected were compared graphically to show whether the Simplex equation can predict the mechanical properties of polymer blends or not. All factors that may effect the mechanical properties will be searched and analyzed to identify the scope and limitation of Simplex equation prediction.

3.2 Experiment on High-Density Polyethylene/Polypropylene blends

3.2.1 Chemicals and Materials

- Decahydronapthalene from Fluka was used as solvent to determine molecular weight of polymer by viscosity method.

- Commercial grades of high-density polyethylene (HDPE) and polypropylene(PP) from Thai Petrochemical Industry CO., LTD. with physical properties listed in Table 3.1 were used as received.

Table 3.1 Physical properties of polypropylene and high-density polyethylene

Property Test Method Unit	Melt Index ASTM D1238 (g/10min)	Density ASTM D1505 (g/cm^2)	Yield Strength ASTM D638 (N/mm^2)	Ultimate Elongation ASTM D638 (%)	Notched Impact Strength DIN 53453 (mJ/mm^2)/(kj/m^2)
GA3750	0.06	0.950	>28	>1600	>14
G2855	0.35	0.955	>30	>1700	>5.5
N3260	1.1	0.956	>32	>1700	>8.0
V1160	15	0.957	>30	>100	>1.8
1102H(PP)	1.8	-	35	-	6($0^{\circ}C$) 2($20^{\circ}C$)

Source : Thai Petrochemical Industry Co.,Ltd.

3.2.2 Equipment

- Twin Screw Extruder: Prism,TSE 16TC (as shown in Figure 3.1)
- Hydraulic compression molding machine: Labtech, LP20, 20 MT (as shown in Figure 3.2)
- Universal Testing Machine: Hounsfield Test Equipment, S-Series (as shown in Figure 3.3)
- Du Pont Thermal Analysis 2100: DSC 2910 (as shown in Figure 3.4)
- Ubbelohde Viscometer PSL(ASTM-IP)
- Oil bath: TAMSON, TV4000 (as shown in Figure 3.5)

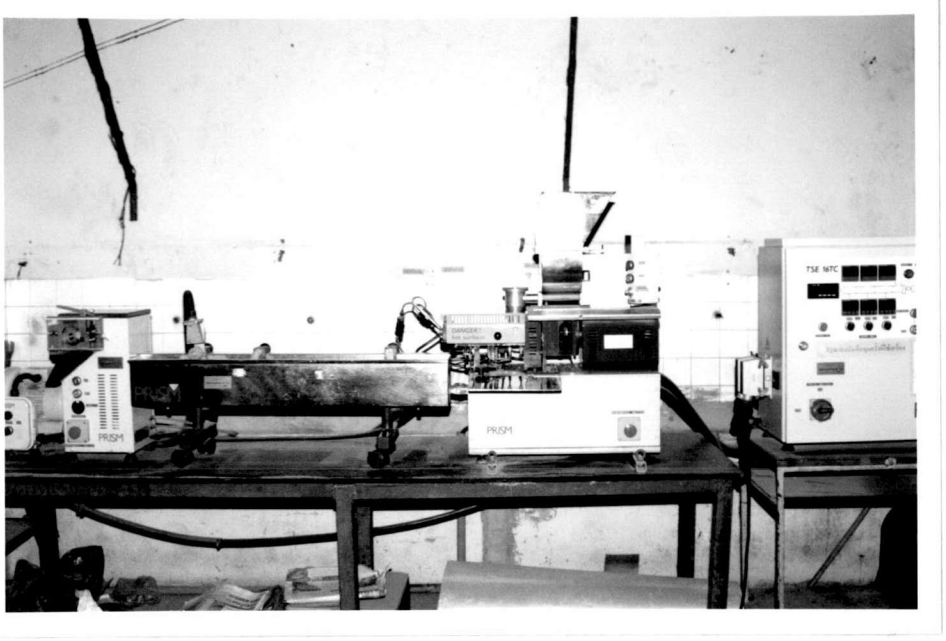

Fig. 3.1 Twin screw extruder

Fig. 3.2 Hydraulic compression molding machine

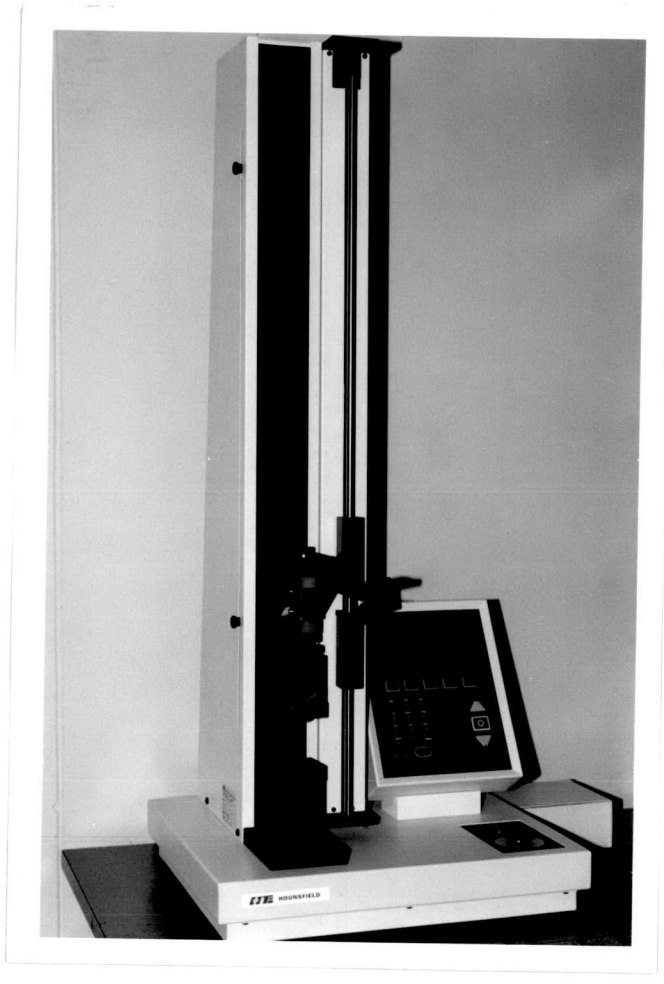

Fig. 3.3 Universal Testing Machine

Fig. 3.4 Differential scanning calorimeter

Fig. 3.5 Oil bath

3.3 Experiment Procedure

3.3.1 Molecular Weight Measurement

Molecular weight of polyethylene and polypropylene as in table 3.1 were determined by the viscosity method, using decahydronaphthalene solutions and calculated from Mark-Houwink-Sakurada equation [32];

$$[\eta] = kM_v^a$$

where

$[\eta]$ = Intrinsic viscosity

k, a = Constant value that can be obtained from the literature for a given polymer-solvent system as in Table 3.2

Table 3.2 Constant value for calculation of average molecular weight by viscosity. [33]

Solvent	polymer	Temperature (°C)	K x 10^3 (ml/g)	a
decahydronepthalene (decalin)	HDPE	135	62	0.70
	PP	135	11.0	0.80

Sample solution preparation [34]

0.40 gram of HDPE sample was transferred to a 100 ml volumetric flask. Approximately 60 ml of decahydronapthalene was added to the flask. The flask was placed in an oil bath maintained at 135°C and shaken once every 10 minutes until HDPE or PP dissolved completely then the solution was added up to 100 ml mark with solvent and maintained at 135°C.

The concentrations of 0.24, 0.16, and 0.08 g/100 ml were made from the above solution (0.40g/100ml)

Typical procedure for viscosity determination [35]

Approximately 15 ml of decahydronapthalene was transferred into an Ubbelohde Viscometer which was securely positioned in the oil bath kept constant at 135°C until the solution attained thermal equilibrium(about 5 minutes). The liquid level was brought to approximately 10 mm above the graduation mark on the viscometer capillary. As the menaces passed this point, the timer was started and the solution was drain to the lower mark on the capillary by intervals. The efflux time of the solution was measured at least three times. Three consecutive readings should agree within 5% for successive flow measurements. The solution was then removed from viscometer.

In the same manner as decahydronapthalene, three consecutive efflux times of HDPE and PP sample solutions were obtained.

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3.3.2 Preparation of HDPE/PP blends

Mixing of PP/HDPE

Polypropylene(1102H) was blended with high-density polyethylene with four difference molecular weight. Seven compositions of 0, 20, 40, 50, 60, 80, and 100% were prepared on a twin screw extruder at 190-210 ^{0}C. The extruder was passed through a water-cooling trough, dried, and subsequently pelletized. Temperature setting in blending of each composition of blends are given in table 3.3.

Table 3.3 Summary of blending condition in twin-screw extruder

Composition		Temperature Setting, ^{o}C		
PP/HDPE	Zone	Feed	Compression	Die
100/0, 80/20, 60/40, 50/50, 40/60, 20/80, 0/100		190	200	210

Preparation of test specimen

Sheets (2.00 mm thick) were obtained by pressing pellets of the blends between metal sheets covered with aluminum foil in a hydraulic press at temperature and time as summarized in Table 3.4 .The sheet were cooled in mold by quenching by cooling water circulated through the plates of the press. Testing specimen were punched out from this sheets into dog bone specimens conforming to ASTM D-638M Type IV for tensile testing.

Table 3.4 Operating condition of Hydraulic Compression Molding Machine.

Blend Composition PP/HDPE	Temperature (^{o}C)	Heating Time (min)	Cooling Time (min)	Pressure (psi)
100/0, 80/20, 60/40, 50/50, 40/60, 20/80, 0/100	210	8	6	2000

Mechanical properties testing

The tensile properties were obtained in accordance with ASTM D638M with the specimen of type IV.(see Appendix A). The operating condition of an Hounsfield Test Equipment, S-Series was as follows:

Cross head speed	25 mm/min.
Distance between Grips	64 mm
Gauge Length	25 mm
Width of narrow section	6 mm
Temperature	25 ^{0}C
Humidity	50 %

All tests were run on identical fashion throughout this work. The only variables were molecular weight and composition. An average $\overline{X}$ of ten specimens was considered as representative value. For any series of tensile measurements, the standard deviation was first calculated via

$$\sigma_s = \left[\frac{\sum_{i=1}^{N}(x_i - \overline{x})^2}{N-1}\right]^{1/2}$$

where x_i represents the i^{th} measurement, $\overline{x}$ the mean of series of measurements, and N the measurement population. The confidence limits for average values are reported as follows:

$$\overline{X} \pm \frac{t\,\sigma_s}{\sqrt{N}}$$

for ten measurement, t= 2.26 at the 95 percent confidence level.

3.3.4 Thermal characterization of polymer blends

Differential Scanning Calorimeter (DSC) was used to characterize crystallinity of these PP/HDPE blends. Samples were heated up to 200^{0}C in nitrogen atmosphere with heating rate of 10^{0}C/min. The degree of crystallinity is calculated by rationing the ΔH thus obtained with that for 100% crystallinity PE (290J/g).