CHAPTER 3

APPARATUS AND EXPERIMENTAL METHOD

Effect of fillers on the mechanical properties of high density polyethylene was studied. To testthe polyethylene and polyethylene filled with calcium carbonate and carbon black at various percentages, the mechanical properties: tensile strength, tensile modulus, elongation, notched Izod impact strength and hardness were measured. Microstructure of the filled polyethylene was observed by a scanning electron microscope.

3.1 Raw materials

3.1.1 High density polyethylene used in this study is identified as Polene grade R-1760, injection molding grade, supplied by Thai Petrochemical Industry Co., Ltd. Some mechanical properties of this polymer are shown in Table 3.1

3.1.2 Fillers

3.1.2.1 Calcium carbonate 1939

3.1.2.2 Calcium carbonate 039

Both carbonate fillers are supplied by Surint Omya Chemicals (Thailand) Co., Ltd. The carbonate fillers properties are shown in Table 3.2

3.1.2.3 Carbon black was supplied by Thai Carbon Black Co., Ltd. Table 3.3 illustrates the properties.

3.1.3 High density polyethylene filled with glass fiber 20% by weight supplied by Thai Petrochemical Industry Co., Ltd. was studied for the purpose of comparison with the high density polynethylene filled with calcium carbonate and carbon black.

Table 3.1 Some properties of polyethylene

properties	test method	unit	value
melt index[2.16 kg/190 C]	ASTM D1238	g/10 min	6.00
melt index[5 kg/190 C]	ASTM D1238	g/10 min	18.00
density	DIN 53479	g/cm^3	0.957
tensile strength	ASTM D 638	N/mm^2	> 14
yield strength	ASTM D 638	N/mm^2	> 30
ultimate elongation	ASTM D 638	%	>250
ball indentation hardness	DIN 53456	N/mm^2	> 54
notch impact strength	DIN 53453	MJ/mm^2	> 3
vicat softening temperature	ASTM D1525	^oC	—
haze	ASTM D1003	% max	—
gloss	ASTM D2457	% min	—
impact strength	ASTM D1709B	g	—

Table 3.2 Some properties of calcium carbonate

Properties	Grade	
	1939	039
Chemical composition		
$CaCO_3$	99.0 %	99.0 %
MgO	0.4 %	0.4 %
SiO_2	0.2 %	0.2 %
Al_2O_3	0.2 %	0.3 %
Fe_2O_3	0.02 %	0.1 %
Physical properties		
Brightness	97.0 %	97.0 %
Moisture content	0.2 %	0.2 %
Specific gravity	2.7 %	2.7
DOP absorption	25 g/100 g	25 g/100 g
Oil absorption	20 g/100 g	20 g/100 g
pH	8	--

Table 3.3 Some properties of carbon black.

Description	Unit
ASTM No.	N330
ASTM type	HAF
DBP Absorption (cc/100g)	102
CTAB (m^2/g)	83
Nitrogen Adsorption (mg/g)	82
Pour density (Kg/m^3)	375
Sieve Residue (%max.)	
U.S.Sieve 35	0.001
U.S.Sieve 325	0.1
300% Modulus(MPa),145^{o}C	-0.2

3.2 Apparatus

3.2.1 Sample preparation apparatus

3.2.1.1 Two roll mixing mill model L.R.M.110 from Lab Tech Engineering Co.,Ltd. with the specification shown in Table 3.4

Table 3.4 The specification of the two roll mixing mill.

Description	Unit
Roll diameter (mm)	110
Roll width (mm)	280
Roll width, working band, (mm)	250
Approximately loading (g)	100-300
Roll pitch (mm)	0.1-2.0
Friction ratio	1:1.2
Front roll speed (rpm)	20
Back roll speed (rpm)	24
Temperature, max, (^{o}C)	300
Temperature rising rate	
room temp. to 200^{o}C with in (min.)	15
room temp. to 300^{o}C with in (min.)	45
Motor power (Kw)	2.2
Electrical system :	3 phase 380-440 volts/50 hertz
	3 phase 220-240 volts/60 hertz

3.2.1.2 Hydraulic hot pressing model LP 20 from Lab Tech Engineering Co.,Ltd. with the specification shown in Table 3.5

Table 3.5 The specification of hydraulic hot pressing

Description	Unit
Compression plate size (mm^2)	200 x 200
Maximum force (Kg)	200
Heating consumption (Kw)	2 x 3
Hydraulic power (Kw)	2.2

3.2.2 Sample testing equipment

3.2.2.1 Tensile tester : Shimadzu AUTOGRAPH model S-100-C, cross head movement 10 mm/min, load selected 50 Kg.

3.2.2.2 Hardness tester: Durometer model QD, testing head shore D from Instrument & Mfg. Co., Inc.

3.2.2.3 Izod-charpy impact tester:Toyo seiki model 612

3.2.2.4 Scanning Electron Microsscope (SEM) model JSM-35 CF JEOL Ltd.

3.3 Experimental procedure

3.3.1 Study of some physical properties of filler

3.3.1.1 Particle size distribution of fillers by sedimentation method.

3.3.1.2 Surface area per gram of fillers by BET method.

3.3.2 Sample preparation

3.3.2.1 The samples were prepared by mixing polyethylene pellet with different weight percents of filler on the two rolls mill for 10 min. Temperatures of the mixing roll were maintained at 150°C (front roll) and at 140°C (back roll). Additional manual mixing by spatula was necessary. After the mixing period, the filled polymer was left on the rolls and cooled down to 80°C then the sample was removed out of the rolls and cut before molding. Hot pressing was at about 150°C after 5 min preheating then pressure was rised up to 2,000 psi for 2 min. The compressed sheet was transfered to the cooling part and cooled about 5 min. The sheet approximately 177 mm x 177 mm x 3 mm was then cut to standard dimensions according to the test methods.

The calcium carbonate filled polyethylene samples were prepared with different amounts of fillers, 2, 5, 10, 15, 20, 25, 30, 35, 40, and 50% by weight of $CaCO_3$ 1939 and 2, 5, 10, 20, 30, and 40% by weight of $CaCO_3$ 039. For carbon black filled polyethylene, the compositions incorporated are 0.5, 1, 2, 5, 10, 20, and 30% by weight. And 20% by weight of HDPE-filled with glass fiber was used as

its original pellet and half diluted to 10% by weight with HDPE resin to form two concentrations of glass fiber-filled polyethylene

3.3.3 Mechanical measurement

The properties of filled polyethylenes were measured according to the following test methods:

3.3.3.1 ASTM D-638 for tensile properties.

3.3.3.2 ASTM D-256 for notched Izod impact strength.

3.3.3.3 ASTM D-2240 for durometer hardness.

3.3.4 Microstructure observation.

The fracture surface from tensile test was studied by observing the microstructure using a Scanning Electron Microscope.