CHAPTER III
EXPERIMETAL

3.1 Materials

The materials used in this study are as follows:

3.1.1 High Density Polyethylene (HDPE)

High density polyethylene of blow molding grade (Marlex HHM 5502) was kindly supplied by Rianthai Interplas Co., Ltd. Some physical properties are shown in Table 3.1

Table 3.1 Physical properties of Marlex HHM 5502 HDPE.

Property	Test method	Unit	Value
Density	D 1505	g/cm^3	0.955
Melt index	D 1238	g/10 min	0.35
Tensile strength at yield(50.8 mm/min)	D 638	MPa	28
Elongation at break (50.8 mm/min)	D 638	%	>600
Shore hardness D	D 2240	-	67
Flexural modulus	D 790	MPa	1380
ESCR, F50	D 1693	Hours	45

3.1.2 Ethylene Gas

The National Petrochemical Public Company Ltd. supplied polymerization grade ethylene gas.

3.1.3 Milled Glass Fiber

Milled glass fiber was purchased from Owen-Corning Co., Ltd. The product description is 731EC 1/32". A summary of the product data is given in Table 3.2.

Table 3.2 Summary of milled glass fiber product data.

Sizing Type	Average filament diameter (μm)	Nominal bulk density (g/cm^3)	Appearance
Cationic	15.8	0.65	Powdery

The glass fibers are relatively free-flowing with a high bulk density. It can be used in formulations which require very uniform dispersions and short fiber lengths.

3.1.4 Sodium Dodecyl Sulfate (SDS)

Sodium dodecyl sulfate (purity 99%+) served as a surfactant to form the surfactant template, or bilayer, for polymerization. It was purchased from Sigma Chemical Co. The material is a white powder, completely soluble in water. A solution of SDS in distilled water was used for all experiments.

3.1.5 Initiator

Sodium persulfate (purity 99%+) acted as a water-soluble initiator. It was purchased from BDH Laboratory Suppliers.

3.1.6 Solvent

Dimethylformamide (AR-grade) was perchased from J.T. Baker Company Limited. It was used for extracting sizing agent from as-received glass fiber using the soxhlet extraction method.

3.2 Instrumentation

3.2.1 Bench Top Parr Reactor

Polymerization reactions were carried out in a 600 mL Parr reactor (Parr Instrument Company), equipped with a pitch-blade turbine impeller, speed controller, external heating jacket, thermowell, and pressure transducer (Figure 3.1).

The impeller speed, outer jacket temperature, internal reactor temperature, and pressure readings are digitally displayed on the front panel.
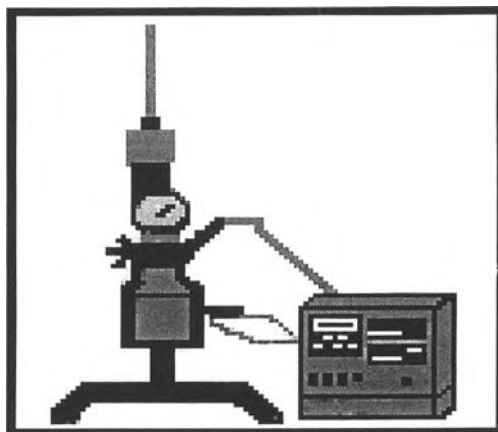

Figure 3.1 Bench-top Parr reactor.

3.2.2 Brabender Plasti-Corder

Each type of glass fiber was mixed with HDPE in a Brabender Plasti-Corder, PL-2100, torque rheometer equipped with a set of removable roller rotors. The measuring mixer W50 had a volumetric capacity of 55 cm^3 and was used in heat and shear stability mode.

3.2.3 Instron Universal Testing Machine

Tensile and flexural tests on the glass fiber reinforced HDPE composite samples were carried out on an Instron Universal Testing Machine, model 4206.

3.2.4 Zwick Pendulum Impact Tester

Impact tests on HDPE/glass fiber composite samples were performed on a Zwick Pendulum Impact Tester, model 5113.

3.2.5 Scanning Electron Microscopy (SEM)

Scanning electron micrographs were taken to identify the morphology of the glass fiber samples. Samples were characterized using a JEOL JSM 5000 scanning electron microscope at a magnification of X3500 and X5000.

3.2.6 Wabash Compression Molder

Test specimens of glass fiber/HDPE composites were prepared by compression molding in a 50 ton capacity Wabash V50H compression press.

3.3 Methodology

3.3.1 Fiber Preparation

Three types of glass fibers were used:

(i) As-received glass fiber – used directly without any further surface treatment.

(ii) Untreated glass fiber – the as-received glass fiber after removal of sizing agent by soxhlet extraction using dimethylfomamide for one day at 150^0C.

(iii) Admicellar-treated glass fiber – surface modified glass fiber (after removal of the original sizing) via admicellar polymerization.

The as-received and modified glass fibers were then washed several times with distilled water and filtered before heating in a muffle furnace at 500^0C for 10 minutes. Then the fibers were washed several times with distilled water, filtered, and dried overnight in a vacuum oven at 80^0C.

3.3.2 Admicellar Polymerization of Ethylene onto Glass Fiber

Untreated glass fiber (25 g) was added to 150 mL of distilled water. The aqueous mixture was adjusted to pH 4 using 0.1 M HCl, then poured into a Parr reactor which was then closed and sealed. After purging the reactor with nitrogen gas, ethylene gas was introduced into the reactor and the pressure adjusted to 150 psi (10.21 atm). For the adsolublization process, the mixture was stirred at 20 rpm for 24 hours at 25^0C. To start the polymerization reaction, the temperature of the mixture was raised to 70^0C and the stirrer speed changed to 60 rpm. Mixing was continued for a further 24 hours to ensure complete polymerization. The change in pressure versus time was recorded at hourly intervals to monitor the ethylene consumption during the reaction. When equilibrium was established, i.e. the pressure remained constant, the reactor was cooled down to room temperature. The

supernatant liquid was carefully decanted from the reactor and the fibers washed several times with distilled water to remove traces of surfactant and initiator. Lastly, the fibers were dried in a vacuum oven at 60^0C overnight.

3.3.3 Surface Morphology of Glass Fibers

Surface morphology of the different kinds of glass fiber samples was characterized using SEM. Samples were mounted on stubs on a sputtering device and sputter coated with gold for 4 minutes. The surfaces of the coated samples were observed by SEM at an accelerating voltage of 25 kV and magnification X3500.

3.3.4 Gravimetric Analysis (Percentage Weight Loss)

This characterization method is based on the difference in weight of the surface-modified glass fibers before and after burning in a furnace at 500^0C for 15 minutes to remove the PE-coated film from the glass fiber surface. The percentage of coating is equal to the percent weight loss which was determined using equation 3.1.

$$\% Wt.\,loss = \frac{Wt.\,of\ glass\ fiber\ before\ burning - Wt.\,of\ glass\ fiber\ after\ burning}{Wt.\,of\ glass\ fiber\ after\ burning}\ 100 \qquad (3.1)$$

3.3.5 Polymer Composite Preparation

The mixing of HDPE with the three types of glass fiber, i.e., as-received glass fiber, untreated glass fiber, and admicellar-treated glass fiber, was carried out using a Brabender Plasti-Corder. The amounts of glass fiber and HDPE used are shown in Table 3.3. A rotor speed of 50 rpm and mixing chamber temperature of 150^0C was used. The batch composition blends were calculated using equations 3.2 and 3.3.

$$D = \frac{M}{V} \qquad (3.2)$$

where:

D = density of the mixed blend

M = mass of the mixed blend

$$V = \{(M_1/D_1)+(M_2/D_2)+(M_3/D_3)+....\}$$

And

$$\text{Batch size (g)} = V_c \times F \times D \qquad (3.3)$$

where:

V_c= volume of chamber

F = fill factor of blending system (0.8).

Table 3.3 Batch composition of each component for glass fiber reinforced HDPE composites.

Glass fiber content (wt%)	HDPE (g)	Glass fiber (g)
20	40.3	10.1

Sheets of composites, 3 mm thick, were prepared by compression molding at 180^0C in a Wabash V 50H compression press. The molding cycle was (a) preheat for 5 minutes (b) compress under a force of 10 tons for a further 3 minutes, and (c) cool under load using cold water and air until the mold reached 40^0C.

3.3.6 Mechanical Properties of HDPE/Glass Fiber Composites

3.3.6.1 Tensile Properties

The tensile strength of the glass fiber reinforced HDPE composites were determined using an Instron Universal Testing Machine. Type I dumbbell specimens were cut out using a pnuematic specimen cutter. Tests were carried out according to ASTM D638 using a 100 kN load cell and 50.80 mm/min cross-head speed. Seven specimens were determined for each sample and the results averaged to obtain a mean value.

3.3.6.2 Flexural Properties

The flexural strength of the composites was measured using an Instron Universal Testing Machine according to ASTM D 790 in three-point bending mode. Measurements were carried out using a 5 kN load cell, 11.52 mm/min cross-head speed and a 43.2 mm support span. Seven specimens were tested for each sample and the results averaged to obtain a mean value.

3.3.6.3 Impact Properties

Izod impact tests were carried out on a Zwick Impact Test instrument using a 2.7 joules pendulum according to ASTM D 256 test method. The results were reported by evaluating a mean value obtained from seven specimens.