

CHAPTER III

EXPERIMENTAL

3.1 Chemicals and Materials

Types and sources of raw materials used in this experiments were shown in Table 3.1.

Polypropylene flakes used in this experimental works were additive-free polypropylene calling (pre-transition of Pro-fax 6331). If the additives were added to modify some properties, it would be called Pro-fax 6331. Some specifications of the polypropylene flakes were shown in Table 3.2. Polypropylene sheet could be formed at high temperature with compression molding technique to provide higher mechanical strength.

Glass fiber used in the research work was chopped strand mat which was suitable for preparation of polypropylene composites. Length of the glass fiber was about 45-50 mm and weight of the fabric was approximately 450 g/m². Due to anisotropic dispersion of the fiber in the glass mat, fiber direction has no significant effect on mechanical properties of the composites.

Surface treating agents used in the experiments were polyvinylalcohol (PVA), anionic polyacrylamide (Fogger), sodium-carboxymethylcellulose (Tyrose[®]), methacryloxypropyltrimethoxysilane (A-174), and maleated PP (Epolene-43P). The role of surface treating agent was to enhance adhesion between dissimilar surfaces. It not only increase

Table 3.1 Materials and Source used in the PP Glass Composites.

Material	Chemical name	Trade name	Source
Resin	Polypropylene flake	Pre-transition of Pro-fax 6331	HMC POLYMER Co.,Ltd.
Reinforcements	Chopped strand mat glass fiber	-	AGI Co.,Ltd.
Surface treating agent	Anionic polyacryl- amide	Fogger	Unitech Co.,Ltd.
	Sodium-carboxy- methyl cellulose	Tyrose [®]	Unitech Co.,Ltd.
	Polyvinyl alcohol	-	ASC Senal Co., Ltd.
	Methacryloxypropyl- trimethoxysilane	A-174 silane	Union Carbide Co.,Ltd.
	Maleated PP	Epolene-43P	White Group Co.,Ltd.
Wax	Mold Release Wax	TR-104	Phol Dhanya Co., Ltd.

Table 3.2 Some Physical Properties of the Polypropylene.

Typical resins properties ^(a)	Pro-fax 6331	ASTM Method ^(b)
Melt flow rate , g/10 min	12	D 1238
Density , g/cm	0.903	D 792 A-2
Notched Izod impact strength at 73°F (23°C), ft-lbs/in (J/m)	0.5(25)	D 256A
Tensile Strength at yield , psi (MPa)	5,000 (34)	D 638
Elongation at yield, %	10	D 638
Flexural modulus, psi (MPa)	235,000(1,620)	D 790B
Rockwell hardness, R scale	97	D 785A
Deflection temperature at 66 psi (455 kPa), °F(°C)	205(96)	D 648
Environmental stress-cracking, hrs	>500, no fail	D 1693

(a) Values shown are averages and are not to be considered specifications.

(b) ASTM test method are latest Society's current procedures.

the true adhesion, but also increases wettability, rheology, and other handling properties.

3.2 Machines and Equipments

The machines and equipments used in this experiment were as follows:

1. Compression molding machine: Hydraulic press type was used for this experiment. Its details are shown in Table 3.3.

2. Mold: The mold was a rectangular shape metal having the following dimensions; 152.4 mm in width, 254 mm in length, and 4 mm in thickness.

Table 3.3 The Details of compression molding machine.

Description	Units	
Clamping force	65	ton
Table size	30 x 45	cm
Stroke	38	cm
Travelling Speed	75	mm/min
Shut Height	100	mm
Heater , automatic control	6000	watt
Motor	15	HP

The compression molding machine was installed with water cooling system and temperature-control devices with 300°C maximum temperature.

3. Low speed cutting machine: The low speed cutting machine used was a 100 mm circular diamond blade saw with adjustable revolution from 0-1,000 rpm.

4. Universal Testing Machine: LLOYD 500

5. Melt Indexer, Model 2050

6. Metallurgical Microscope, Model BHSM-313B

7. Thermocouple and Digital thermometer: Digicon DP 500, (-50 to 750°C)

8. Electronic analytical balance

9. Vernier caliper

10. Stop watch

11. Porcelain Crucibles

12. Electric Furnace

3.3 Determination of the Optimum Condition for Glass Fiber Reinforced Polypropylene Preparations

In the processing of glass fiber reinforced polypropylene, the factors affected the flexural properties may consist of pressure at melting period, pressure at cooling stage, holding time at melt and melting temperature so such factors have to be optimum.

3.3.1 Determination of the Optimum Pressure at Melting Period

Experimental of determination of the optimum pressure at melting period was performed as following.

1. The glass fiber was cut into a size of 6 x 10 inches for three layers and their weight was determined.

2. The polypropylene flakes were mixed with the fiber at the weight ratio of 70 to 30.

3. The mold temperature was set approximately at 200°C.

4. The mold surfaces were cleaned by paint brush and were treated with mold releasing agent (TR-104, High Temperature grade).

5. Three layers of the fiber were placed in the mold alternately with four layers of the polypropylene flakes.

6. The mold was closed and kept the pressure at 0 psi. The sandwich was held until the temperature reached 200 °C and held further for 5 minutes.

7. The pressure was adjusted to 1000 psi and then the sandwich was cooled for 30 minutes.

8. The mold was opened and removed from the hydraulic press.

9. The procedures 1 to 7 were repeated but the holding pressure in step 7 was changed to 200, 500 and 1000 psi.

10. For a typical cycle time, the molding process usually requires the following condition:

Sandwich preparation	18	sec
Molding closing time	5	sec
Melting time	3,600	sec
Cooling time	1,800	sec
Mold opening time	7	sec
Mold removal	7	sec
Total time	5,437	sec

11. Relationship between flexural properties of the products and the pressure at melt was determined.

3.3.2 Determination of the Optimum Pressure at the Cooling Stage

All procedures in Section 3.3.1 were repeated except step 7, the pressure at cooling state was changed to 0, 200, 500, and 1500 psi. The relationship between flexural properties of the products and the pressure at the cooling stage was determined.

3.3.3 Determination of the Optimum Holding Time

All procedure in Section 3.3.1 were repeated except step 7, the holding time at melt was varied to 0, 10, 20 and 30 min. The relationship between flexural properties of the products and the holding time at melt was determined.

3.3.4 Determination of the Optimum Melting Temperature

All procedures in Section 3.3.1 were repeated except the pressure at melting period was changed to 500 psi and holding at 0 psi in step 6 was eliminated. The melting temperature was changed to 180, 220, and 240°C, respectively. The relationship between flexural properties of the products and the melting temperature was determined.

3.3.5 Determination of the Optimum Fiber Content

Experimental determination of the optimum fiber content was performed as following.

1. All procedures in Section 3.3.1 were repeated except the weight ratio of polypropylene flakes to glass fiber weight was varied from 20 to 50% fiber content.

2. The optimum condition used in the experiment is shown below.

Temperature	200	°C
Pressure at melt	0	psi
Pressure at cool	500	psi
Holding time	0	minute

3. Relationship between flexural properties and glass fiber content was determined.

3.4 Investigation of the Effect of the Surface Treating Agent on Flexural Properties

Experimental works concerned with comparison of the flexural strength of glass fiber reinforced polypropylene when the glass fiber was coated and was not coated with coupling agent.

3.4.1 Effect of the Surface Treating Agent together with the Coupling Agents on Commercial Glass Fiber

The surface treating agents used in this experiment were water soluble except Epolene-43 P which was dissolved in hot toluene.

3.4.1.1 Water Soluble Surface Treating Agents

Surface treating agent of glass fiber with water soluble surface treating agents for preparation of the composite would consist of:

1. The glass fiber mat was cut into a size of 6 x 10 inches.
2. The 0.10% (weight by volume) aqueous solution of PVA, Tyrose[®], Fogger, and A-174 silane were prepared and poured into plastic trays separately.
3. The fiber mats were soaked into the prepared solutions separately, at room temperature for 2 days and the trays were covered with a glass plate.
4. The fiber mats were removed from the soaking solu-

tions and dried in an oven at 70°C for 12 hours.

5. All procedures in Section 3.3.1 were repeated with the following condition.

Temperature	200	°C
Pressure at melt	0	psi
Pressure at cool	500	psi
Holding time	0	minute
Glass fiber content	35%	(by weight)

6. The surface treating agent in procedure 2 was changed according to Tyrose[®], Fogger, and A-174, respectively.

7. Relationship between flexural properties of the products and type of surface treating agent was determined.

3.4.1.2 Toluene Soluble Surface Treating Agents

Surface treating agent of glass fiber with toluene soluble surface treating agents for preparation of the composite would consist of:

1. The glass fiber mats were cut into a size of 6 x 10 inches.
2. The 0.10% (weight by volume) solution of Epolene-43P in hot toluene was prepared and poured into an aluminium tray.
3. The cut glass fibermats were soaked in the hot solution at room temperature for 5 minutes.
4. The fiber mats were removed from the solutions,

washed with acetone and then dried in an oven at 120°C for 3 hours.

5. All procedures in Section 3.1.1 were repeated with the following condition in step 5 of section 3.4.1.1.

6. Flexural strength of the products was determined.

3.4.2 Effect of the Surface Treating Agents Coated on Coupling Agents Free-Glass Fiber

Experimental procedure for investigation of effect of the surface treating agents coated on coupling agents free-glass fiber was performed as following:

1. The glass fiber mat was cut into a size of 6 x 10 inches and burned in an electric furnace at 550°C for 5 hours.

2. All procedures in Section 3.4.1.1 and 3.4.1.2 were repeated.

3. Relationship between flexural properties of the products and types of surface treating agent were determined.

3.5 Estimated Polynomial Regression Equation

The flexural strength and modulus were determined with enclosure of the effect of glass fiber content and percent of void by using of polynomial regression equation method in computer software of S.P.S.

3.6 Flexural Testing Procedure

The flexural test described below was based on the test method of ASTM D 790-90.

1. The specimens were cut for flexural testing with the low speed cutting machine in a size of 25 mm of width, 80 mm of length, and approximately 4 mm of thickness and the rim was trimmed with a cutter.

2. For a 3-point bending flexural testing, the length of the support span depends upon the thickness. In this case 50 mm span length was used in order to have a support span-to-thickness ratio of 16:1 as shown in Figure 3.1. A test specimen was loaded via a 10.1 mm diameter of crosshead loading nose by using the rate of crosshead motion at 2.00 mm/min.

3. Flexural strength and flexural modulus were calculated by the following equations:

$$S = 3PL / 2bd^2 \quad (3.1)$$

$$E_b = L^3 m / 4bd^3 \quad (3.2)$$

where

S = strength of bending at mid span (MPa, N/mm²)

E_b = modulus of elasticity in bending (MPa, N/mm²)

P = load at the given point on the load-deflection curve(N)

L = length of support span (mm)

b = width of beam tested (mm)

- d = depth of beam tested (mm)
- m = slope of the tangent to the initial straight-line portion of the load-deflection curve (N/mm)

3.7 Fiber and Void Content Determination

The experiments described below was based on the test methods of ASTM D 2584-68, ASTM D 792-91 and ASTM D 2734-91.

3.7.1 Determination of the Glass Fiber Content

1. The specimens were cut along the deflection line of the bending test with the low speed cutting machine in a size of 10 mm of width and 25 mm of length and the rim was trimmed with a cutter.
2. The specimens were weighed and placed in a crucible and then ignited with a bunsen burner.
3. When the burning ceased and remained only ash and carbon, the crucible and residue were heated in a furnace at 550 °C until all carbonaceous material had disappeared.
4. The crucible was cooled at room temperature in a desiccator and the weight was determined.
5. The ignition loss of the specimens in weight percent was calculated by the following equation:

$$\text{Ignition loss, weight \%} = [(W_1 - W_2) / W_1] \times 100 \quad (3.3)$$



where

W_1 = weight of specimen (g)

W_2 = weight of residue (g)

3.7.2 Determination of the Void Content

1. The specimens were cut by the low speed cutting machine in a size of 10 mm of width and 25 mm of length.

2. The specimens were weighed in air and water. The density of the specimens was calculated.

3. All procedures in Section 3.6.1 were repeated. The resin content and the glass content (% weight) were calculated.

4. The void content was calculated by the following equation:

$$V = 100 - M_d[(r/d_r) + (g/d_g)] \quad (3.4)$$

where

V = void content (volume %)

M_d = measured density

r = resin (weight %)

g = glass (weight %)

d_r = density of resin

d_g = density of glass

3.8 Investigation of the Effect of the Holding Time on Molecular Degradation by Using of the Melt Flow Rate

Experimental works concerned with comparis of the melt flow rate at any holding time used in the process.

3.8.1 Preparation of the Sample

1. The mold temperature was set approximately at 200°C.
2. The mold surface was cleaned by paint brush and was treated with mold releasing agent(TR-104, High Temperature Grade).
3. The polypropylene flakes were placed in the mold and then the mold was closed and kept the pressure at 0 psi.
4. The sample was held until the temperature reached 200°C and then the pressure was adjusted to 200 psi. After that, the sample was cooled for 30 minute.
5. The mold was opened and removed from the hydraulic press.
6. All procedures 1 to 5 were repeated but the holding time at melt was varied to 5,10,20 and 30 minutes.
7. The sample were scraped with circular cutter and kept a sample for determination of the melt flow rate (section 3.8.2).

3.8.2 Determination of the Melt Flow Rate by Melt Indexer

The experiment described below was based on the test method of ASTM D 1238-90b. This method covers the measurement of the

rate of extrusion of molten resins through a die under prescribed conditions of temperature, load, and piston position in the barrel as the time measurement is being made. The values stated in SI units are to be regarded as the standard. Experimental procedure was performed as following.

1. The conditions of temperature and load used in this experiment were 190°C and 2.16 kg, respectively.
2. The apparatus were cleaned. The parts can be cleaned easily while they are still hot.
3. The piston was removed and placed on an insulated surface.
4. The cylinder was changed with Teflon funnel with 6 g of sample. The sample was compacted as tight as possible using a compaction tool put through funnel. This procedure removes most of the air.
5. The loaded piston was placed on top and allowed a minimum of 6 minutes for preheat time.
6. The loaded piston was placed on top. After 30 second, a scribe line was reached the guide bushing and cut off the melt by a spectula and then the timer was started simultaneously to measure again.
7. The extrudate was weighed and multiply the weight by the factor shown in Table 3.4 to obtain the melt flow rate in grams per 10 minutes.

Table 3.4 Standard Test Conditions Sample Weight and Testing Time

Flow Range (g/10 min)	Suggested Weight of Sample in Cylinder (g)	Time Interval		Factor For Obtaining Flow Rate in g/10 min
		Min.	Secs.	
0.15 to 1.0	3	6.00	360	1.67
1.0 to 3.5	5	3.00	180	3.33
3.5 to 10	6	1.00	60	10.00
10 to 25	7	0.50	30	20.00
25 to 50	8	0.25	15	40.00

3.9 Study of Microstructure of the Composites

3.9.1 Preparation of Specimens

1. The specimens used to test bending were cut with the low speed cutting machine in a size of 10 mm of width and 25 mm of length and trimmed the rim with a cutter.

2. The two specimens were placed in a polyethylene cup in the form of V shape.

3. Unsaturated polyester were mixed with a few drops of hardener and then poured in a cup of specimens.

4. The bulk of unsaturated polyester was leaved for curing at room temperature for 2 hours.

5. The specimen was released from a cup after that it was placed in oven at 70°C for 3 hours for post-curing.

6. The surface of specimen was abraded with abrasive paper No. 400, 600, 800 and 1200 respectively, and polished with alumina powder having particle size of 1.0, 0.3 and 0.05 μ m respectively.

7. The microstructure of specimens were studied with Metallurgical Microscope, Olympus Model BHSM-313B and photographed.