CHAPTER IV

EXPERIMENT

4.1 Materials, Chemicals and Equipments

Materials

PA6 resin (1015B), PA6 compound with 15 wt% glass fiber (1015GC3) and 30 wt% glass fiber (1015GC6) were provided by UBE Nylon (Thailand) Limited.

Chemicals

The three chemical substances in sorrogate gasohol are isooctane, toluene and aggressive ethanol. Aggressive ethanol used in this study met SAE J1681 criteria. Aggressive ethanol comprised of ethanol 816.0 g, de-ionized water 8.103 g, sodium chloride 0.004 g, sulfuric acid 0.021 g and glacial acetic acid 0.061 g (for 1 litre of aggressive ethanol). The chemicals required for experiment were obtained from S.R. Labolartory as follows:

- Ethanol (AR Grade, J.T. Baker SOLUSOR)
- Isooctane (AR Grade, BRIGHT CHEM SDN BHD)
- Tolulene (AR Grade, J.T. Baker SOLUSOR)
- Sodium chloride (AR Grade, Merck Chemical)
- Sulfuric acid (AR Grade, Merck Chemical)
- Glacial acetic acid (AR Grade, Merck Chemical)

Equipments

- Vacuum oven (Lab-instrument 3606-1CE)
- Injection molding (Manumold 45E)

- Compression molding (Lab Tech Engineering)
- Universal testing machine (Instron 5567)
- Grinder machine (Buehler Metaserv)
- Notching machine
- Trimming Machine (George Machine CY 135A)
- Digital micrometer
- Balance (Mettler Toledo AG 204)
- Digital vernier calipers
- Dynamic Mechanical Analysis (NETZSCH DMA 242 C)
- HDT/vicat
- Impact Tester (Yasuda)

4.2 Test Specimens Preparation

Both PA6 resin and PA6 compound were dried in a vacuum oven at 80 °C for 24 hour to remove the moisture [11]. The property testing required five different shaped test specimens including

- Disk
- Bar
- Rectangular
- Dumbbell
- Izod

4.2.1 Compression Molding

Disk specimens were prepared by compression molding machine (see Figure 4.1). These test specimens conform to ASTM D570 for physical properties testing.

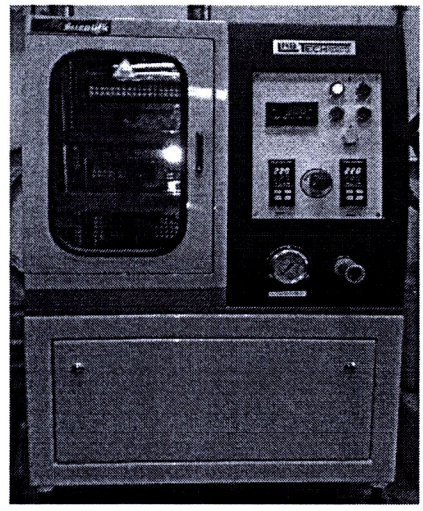

Figure 4.1 Compression molding machine

Table 4.1 Process parameters setting of compression molding machine for PA6

Procoess Parameters	Setting
Mold temperature (°C)	225
Preheating time (min.)	2
Heating time (min.)	4
Cooling time (min.)	6
Molding pressure (bar)	90

Table 4.2 Process parameters setting of compression molding machine for PA6 compound with 15 wt% glass fiber

Procoess Parameters	Setting
Mold temperature (°C)	230
Preheating time (min.)	3
Heating time (min.)	4.5
Cooling time (min.)	6
Molding pressure (bar)	20-90

Table 4.3 Process parameters setting of compression molding machine for PA6 compound with 30 wt% glass fiber

Procoess Parameters	Setting
Mold Temperature (°C)	235
Preheating Time (min.)	4
Heating Time (min.)	3.5
Cooling Time (min.)	6
Molding Pressure (bar)	20-90

4.2.2 Injection Molding

Dumbbell, bar and rectangular shapes were prepared by injection molding machine (Manumold). These test specimens conform to ASTM D638 and ASTM D790 for tensile testing and flexural testing, respectively. The izod and rectangular shapes specimens were cut from the bar shapes that were prepared by injection molding using trimming machine (Band saw) and rubbed by grinder machine. These test specimens conform to ASTM D695, ASTM D7028 and ASTM D648 for compressive testing, dynamic mechanical analysis (DMA) and heat distortion temperature testing, respectively. While izod shapes were notched by notching machine. These test specimens conform to ASTM D256 for impact testing.

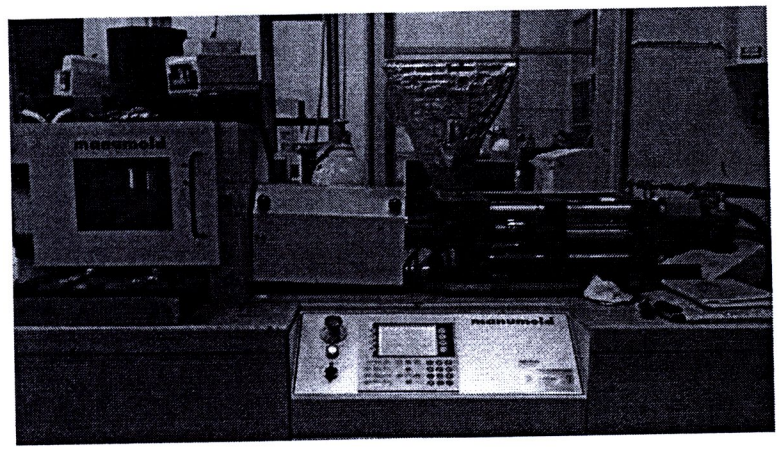

Figure 4.2 Injection molding machine

Table 4.2 Process parameters setting of injection molding machine for PA6

Material	Procoess Parameters		Setting
PA6	Temperature (°C)	Nozzle	290
		Zone 1	285
		Zone 2	285
	Pressure (bar)	Injection forward I	60
		Injection forward II	36
		Injection forward III	36
		Holding	20
		Metering	60
	Stroke (mm)	Injection forward I	25
		Injection forward II	18
		Injection forward III	15
		Holding	15
		Metering	50
	Time (sec)	Holding	3
		Cooling	10

Table 4.3 Process parameters setting of injection molding machine for PA6 compound with 15 wt% glass fiber

Material	Procoess Parameters		Setting
PA6 compound with 15 wt% glass fiber	Temperature (°C)	Nozzle	295-300
		Zone 1	290-295
		Zone 2	290-295
	Pressure (bar)	Injection forward I	65-70
		Injection forward II	40-45
		Injection forward III	40
		Holding	30
		Metering	60
	Stroke (mm)	Injection forward I	25
		Injection forward II	18
		Injection forward III	15
		Holding	15
		Metering	50
	Time (sec)	Holding	2-4
		Cooling	7

Table 4.4 Process parameters setting of injection molding machine for PA6 compound with 30 wt% glass fiber

Material	Procoess Parameters		Setting
PA6 compound with 30 wt% glass fiber	Temperature (°C)	Nozzle	320-325
		Zone 1	315-320
		Zone 2	315-320
	Pressure (bar)	Injection forward I	70
		Injection forward II	45
		Injection forward III	40
		Holding	30
		Metering	60
	Stroke (mm)	Injection forward I	25
		Injection forward II	18
		Injection forward III	15
		Holding	15
		Metering	50
	Time (sec)	Holding	2
		Cooling	5

4.3 Preparation of Chemicals in Surrogate Gasohol

The chemical substances in surrogate gasohol including ethanol, isooctane and toluene were obtained from S.R laboratory and aggressive ethanol is a worst-case-scenario fuel that would still be acceptable under ASTM D4806, *Standard specification for denatured fuel ethanol for blending with gasoline for using as automotive spark-ignition engine fuel.* Formulations of aggressive ethanol components to make 1.0 L are:

- Synthetic ethanol 816.00 g
- De-ionized water 8.103 g
- Sodium chloride 0.004 g
- Sulfuric acid 0.021 g
- Glacial acetic acid 0.061 g

4.4 Test Procedures

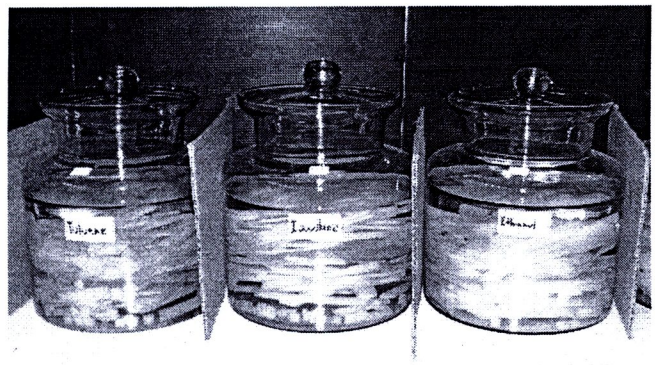

Figure 4.3 Test container

To study the effect of chemical substances in surrogate gasohol on the physical, mechanical and thermal properties of samples, the specimens were immersed in each chemical substance (see Figure 4.3), i.e., ethanol, isooctane, toluene and aggressive ethanol, in separated jars at room temperature. The chemical substances were changed every six weeks. Before physical, mechanical and thermal testing, the specimens would be taken out from the chemical substances and dried in air for 2 h before testing. Physical and mechanical properties were measured in 0^{th}, 1^{st}, 2^{nd}, 3^{th}, 5^{th}, 7^{th}, 10^{th}, 13^{th} and 16^{th} week and thermal properties were measured in 0^{th}, 2^{nd}, 5^{th}, 10^{th} and 16^{th} week.

4.5 Physical Properties Measurement

The disk shaped specimens were used for physical property testing. Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.5.1 Water Absorption

The specimens were immersed in DI water at room temperature. For water absorption testing the specimens would be taken out from the DI water, wiped dry with a lint free cloth and dried in air for 2 h before testing. Balance would be used for weighing of specimens. Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.5.2 Mass and Volume Change Testing

The specimens were immersed in chemical substance at room temperature. For mass and volume change testing the specimens would be taken out from the chemical substances, wiped dry with a lint free cloth and dried in air for 2 h before testing. Balance, Digital vernier calipers and Digital Micrometer, would be used for measuring mass change, diameter change and thickness change, respectively. Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.6 Mechanical Properties Measurement

4.6.1 Tensile Property Measurement

Universal testing machine (Instron model 5567) would be used for tensile property testing. The dumbbell shaped specimens were tested in tension mode at constant crosshead speed of 50 mm/min with a 30 kN load cell at 25 °C according to ASTM D638. Five specimens were tested to determine the average value. From the data, the tensile modulus and the tensile strength of PA6 and PA6/GF composites were obtained.

4.6.2 Flexural Property Measurement

The bar shaped specimens were measured to conduct the flexural strength and flexural modulus. The three point bending tests were done according to ASTM D790. A support span-to-dept ratio was 16:1 and constant crosshead speed of 1.2 mm/min was performed in a universal testing machine (Instron model 5567) with a 30 kN load cell at 25 °C. The loading point for the test was located at the center. Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.6.3 Compressive Property Measurement

The compression test of specimens before and after an immersion in chemicals of surrogate gasohol was performed according to the ASTM D695 test standard on a universal testing machine (Instron model 5567) with a 30 kN load cell at 25 °C controlling constant crosshead speed of 1.2 mm/min. Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.6.4 Impact Property Measurement

The standard test method that describes the Izod impact test is ASTM D256. Izod impact strength of specimens before and after an immersion in chemicals of surrogate gasohol was determined by Impact Tester using acutely notched specimens (notch depth is 2 mm). Five specimens of PA6 and PA6 compounds were tested to determine the average value.

4.7 Thermal Properties Measurement

4.7.1 Glass Transition Temperature

The rectangular shaped specimens were used to measure the glass transition temperature (T_g). The dynamic mechanical properties of specimens before and after an immersion in chemicals of surrogate gasohol were determined by using Dynamic Mechanical Analysis (DMA) in a three-point bending mode with a 110 mN static force and a 110 mN dynamic force. Each specimen was first cooled under liquid nitrogen to -50 °C and then heated at 5 °C/min to 200 °C at a frequency of 1 Hz under nitrogen. The storage modulus (E'), loss modulus (E") and mechanical loss factor tan δ were recorded as a function of temperature. The glass transition temperature of the specimen was obtained from the maximum peak of the loss tangent plot.

4.7.2 Heat Distortion Temperature

The heat distortion temperature of the specimens was measured according to ASTM D648 by HDT/vicat. The specimens were tested in a flatwise position. For the HDT measurements, the specimens were immersed into a silicone oil bath and heated from

room temperature to 200 °C at a heating rate of 2 °C/min. A load of 1820 kPa was applied to the specimen at the center. Once the specimen was deflected by 0.25 mm, the temperature was noted as heat distortion temperature (HDT) of the specimen. Three specimens of PA6 and PA6 compounds were tested to determine the average value.