CHAPTER III

APPARATUS AND EXPERIMENTAL PROCEDURES

In the study of wood flour filled polypropylene composites, the effects of wood flour filler and coupling agents on the mechanical properties of polypropylene were studied. The problem of compatability between the polar wood filler and nonpolar polymer can be overcome by modifying with the conventional coupling agents. Coupling agents were organofunctional silanes and Epolene wax. Microstructure of filled polypropylene was observed by scanning electron microscope.

3.1 Raw Materials and Reagents

3.1.1 Polypropylene

The polypropylene used in this study was all powdered, commercial-available materials: POLENE N-700P, injection moulding grade and processing temperature at 190-240 ^{o}C. It was supplied by Thai Petrochemical Industry Co., Ltd. Typical data of POLENE N-700P are shown in Table 3.1.

Table 3.1 Typical Data of POLENE N-700P.

Properties	Test method	Value
MFI 230/216 (g/10 min)	DIN 53735	
	ASTM D1238	11
Tensile strength at yield (N/mm^2)	DIN 53455	35
Modulus of elasticity		
in tension (N/mm^2)	DIN 53457	1550
Impact strength (kJ/m^2)	DIN 53453	
at 0°C		18
at -20°C		12
Notched impact strength (kJ/m^2)	DIN 53453	
at 23°C		3.5
at -20°C		1.6
Heat distortion temperature (°C)		
at 0.46 N/mm^2	DIN 53461	95
at 1.85 N/mm^2	ASTM D648	60

3.1.2 Wood Flour

Wood flour obtained from sawdust was a waste in the wood manufacturing. The coarse sawdust that was further sieved in order to keep the particle size at smaller than 60 mesh (250 μm) was used in all experiments. Its color is light brown and density is in range of 1.42-1.43 g/cm^3. Some properties of wood flour are presented in Table 4.1.

3.1.3 Coupling Agents

3.1.3.1 Organofunctional Silanes

Two types of organofunction silane were used as: a) γ-Methacryloxy propyltrimethoxysilane (A-174), and b) γ-Ammino-propyltriethoxysilane (A-1100). Both organofunctional silanes were supplied by Union Carbide Corporation. These coupling agents are clear liquid. Chemical structure and some detailed properties of these silane coupling agents have already described in Table 2.7. The physical properties of silane A-174 and A-1100 are shown in Table 3.3.

Table 3.3 Typical Physical Properties of Silanes A-174 and A-1100.

Properties	A-174	A-1100
Formular molecular weight	248.4	221.4
Physical form	CL(c)	CL
Viscosity (cSt at 25°C)	2	2
Apparent specific gravity (25°C/25°C)	1.045	0.946
Refractive index (25°C)	1.429	1.420
Flash point (°C)	88	96
Boiling point (°C)	255	217

cSt = centistrokes

CL = Clear Liquid, (c) = light straw

3.1.3.2 Epolene E-43P Wax

Epolene E-43P is a commercial name of maleated polypropylene of Eastman Chemical Products, Inc., which was supplied by the White Group Limited. It is light yellow powder. The properties of Epolene E-43P wax are summarized in Table 3.4.

Table 3.4 Typical Properties of Epolene E-43P Wax.

Properties	Value
Ring and ball softening point (^{o}C)	157
Density (g/cm^3 at 25^{o}C)	0.934
Acid number	47
Brookfield Thermosel Viscosity[a] (cP)	
at 140^{o}C	solid
at 150^{o}C	solid
at 190^{o}C	400
Color, Gardner scale	11
Molecular weight, GPC	
$\bar{M}_w$	9100
$\bar{M}_n$	3900

cP = centipoise

a = Conventional Brookfield viscosity

3.1.4 Reagents

1) Carbon tetrachloride, AR grade, MERCK.

2) Benzoyl peroxide, Gr grade, MERCK.

3) Xylene, AR grade, CARBO ERBA.

4) Diethyl ether, AR grade, M&B.

3.2 Apparatus and Equipment

1) Two roll mill: LRM 110, Lab Tech. Engineering Co., Ltd.

2) Compression moulding: LP 20, Lab Tech. Engineering Co., Ltd.

3) Universal tesing machine: Instron 4206-006, 100 KN.

4) Impact testing machine: Cantilever beam impact machine (Izod Type), Yasuda Seiki Seisakusho Ltd.

5) Hardness tester: Durometer Type D, Test Unit 7206, ZWICK.

6) Scanning electron microscope: JSM T-220A and JSM T-20, JEOL Co., Ltd.

7) Test sieve: Sonic Sifter A-1, ASAHI (80,100,150 mesh). Endecotts LTD. (60 mesh).

8) Drying oven: SANYO (TSE).

9) Motor control: RW 20, Janke & Kunkel GmbH & Co. KG.

10) Heating mantle: HRE/100, HORST GmbH.

3.3 Experimental Procedures

3.3.1 Wood Flour Analysis

1) The particle size of wood flour was determined by using Sonic Sifter A-1 instrument. In making such an analysis, a set of standard screens was arranged serially in a stack, with the smallest mesh (150 mesh) at the bottom and larger meshes (100, 80 mesh) on the top. Weighed wood flour was placed on the top screen and the stack was shaken mechanically for about 15 min. The particles retained on each screen were removed and weighed, and the wood flour masses of each individual screen were converted to percentage of the total sample. The particle size distribution curve was then constructed.

2) Composition of wood was determined by following TAPPI standard methods.

3) Surface characteristic of wood flour was studied by means of scanning electron microscope.

3.3.2 Treatment of Organofunctional Silane on Wood Flour

1) Oven dried wood flour (50 gram) was placed in a flask to which 300 cm^3 of carbon tetrachloride was added, followed by the addition of benzoyl peroxide (2%) and 2% of silane A-174 or A-1100 in due experiment.

2) The above mentioned mixture was refluxed for 3 hours. After cooling, the carbon tetrachloride was evaporated and the wood flour was dried at $60^{o}C$.

3.3.3 Preparation of the Composites

In this experiment, four different composites were defined as follows.

Untreated : The composites were composed of untreated wood flour and polypropylene.

Epolene E-43P: The composites were composed of untreated wood flour, polypropylene and Epolene E-43P.

A-174 or A-1100 : The composites were composed of treated wood flour with silane A-174 or A-1100 and polypropylene.

3.3.3.1 Effect of Epolene E-43P Concentration on Mechanical Properties of Composite

The composites were prepared at 20% of the untreated wood flour and different weight percents of Epolene E-43P (1 to 5 by weight) were then added.

Compounding

First, wood flour of different percentages was mixed with the powdered polypropylene. Then, the mixture was preheated on the two roll mill for about 5 min. Next, the compounding of polypropylene and wood flour were homogenized on two roll mill for 10 min. The temperatures of the mixing rolls were maintained constant at 168 ^{o}C for the front roll and 158 ^{o}C for the back roll. During the

mixing period, a brass scraping knife and a wood-scrapper were necessary for manual mixing so as to increase good homogeneity in all directions. Finally, the mixture was removed out of two roll mill and folded to a square shape.

Moulding

The wood flour-polypropylene mixture was placed in a mould whose dimension was 150 mm x 160 mm x 32 mm. Then, it was preheated on the heating part for about 10 min. Compression moulding of the composite was done at 200°C. The moulding time was 15 min and the pressure used was 150 kg/cm^2. Finally, the compressed sheet was transferred to the cooling part and cooled for 5 min with the constant pressure at 150 kg/cm^2 during the process. The sheet composite was cut to the standard specimens in according to the ASTM test method

3.3.3.2 Effects of Different Coupling Agents and Filler Content on Mechanical Properties of Composite

In the case of untreated wood flour and treated wood flours with silane A-174 or silane A-1100, the compositions incorporated were 10, 20, 30, 40 and 50% by weight of the filler.

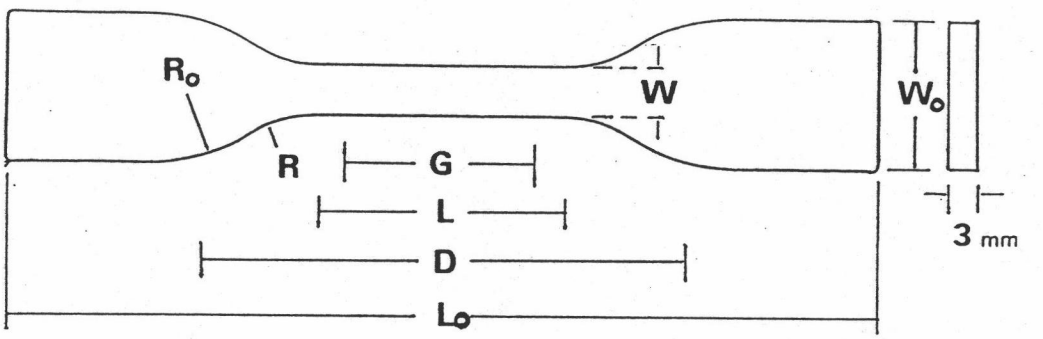
In the case of Epolene E-43P coupling agent, the composites were prepared with different amount of 10, 20, 30, 40, and 50% by weight of the fillers and then 2% Epolene E-43P (by weight of the wood flour) was added.

3.3.4 Mechanical Testing

Mechanical properties of the composites were measured by following the ASTM test methods:

ASTM D638-90: Standard test method for tensile properties.

The test specimens (type IV) dimension was presented in Figure 3.1.



W :	6 mm	W_o:	19 mm	G :	25 mm	R :	14 mm
L :	33 mm	L_o:	115 mm	D :	65 mm	R_o:	25 mm

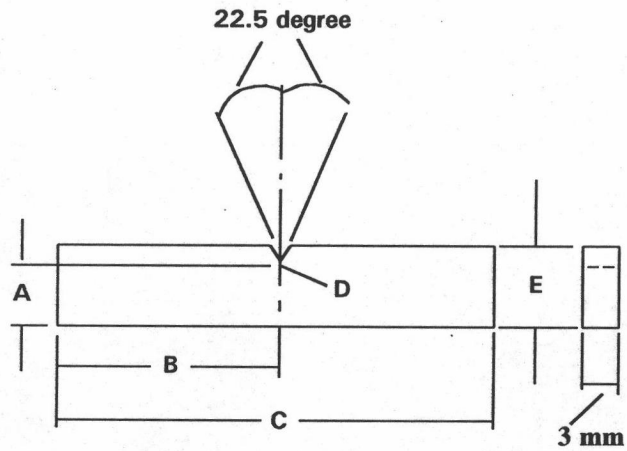
Figure 3.1 Schematic of tensile test specimen (type IV).

The tensile testing conditions were as follows:

Temperature:	25	^oC
Relative humidity:	50	%
Speed of testing:	5	mm/min
Distance between grips:	64	mm
Gage length:	25	mm

ASTM D256-90b: Standard test method for impact resistance.

The test specimens dimension for Cantilever Beam (Izod-type) test is shown in Figure 3.2.



unit : mm

A : 10.16 ± 0.05 C : 63.50 max, 53.50 min

B : 32.00 max, 31.50 min D : 0.25 ± 0.05

E : 12.70 ± 0.15

Figure 3.2 Schematic of Izod type test specimen.

The machine parameters and testing conditions of the impact test were listed below:

Temperature:	25	^{o}C
Relative humidity:	50	%
Pendulum capacity:	11.0	J
Depth of specimen:	10.16	mm

ASTM D2240-91: Standard test method for hardness.

For the assignment of the specimen for hardness testing, the test specimen should be of at least 6 mm in thickness. The surface of the specimen should be flat and parallel over a sufficient area to permit the presser foot to contact the specimen. For materials having hardness values above 50 Type D durometer, the thickness of the specimen should be of at least 3 mm and measurements should not be made closer than 6 mm to any edge.

According to the thickness assignment, the test specimens in this experiment were composed of plied pieces to obtain the necessary thickness. The Type D durometer was used in this experiment. The conditions in testing were shown as follows:

Temperature:	25	^{o}C
Relative humidity:	50	%
Number of pieces plied:	2	pieces

All of the properties measured, at least five samples were tested to obtain a reliable average and standard deviation.

3.3.5 Microstructure of the Fracture Surface of the Composites

The fracture surfaces from tensile testing specimens were observed by a JEOL T-220A scanning electron microscope, operated at 20 KV. The samples were coated with gold before scanning observations.

3.3.6 Examination of the Polarity in Wood Flour Residue

The wood flour residue samples were obtained from the processed composites by a hot xylene extraction. First, the composite sample was cut to small pieces, boiled with hot xylene until the polymer matrix was dissolved in xylene. Next, the wood flour was separated and continuously eluted with new hot xylene for 8 hours. Thus, approximately 0.5 gram dried residue sample was shaken vigorously in the ethyl ether-water mixture and allowed to stand overnight.

3.3.7 Differential Thermal Analysis (DTA) of the Composites

All types of composite were investigated with a Shimadzu Thermal Analysis Instrument DT-30, operated on Differential Thermal Analysis mode from room temperature to 200 ^{o}C, heating rate at 10 ^{o}C/min, sensitivity at ±50 μV, under the N_2 atmosphere and in the opened crucible.