CHAPTER II

EXPERIMENTAL

2.1 Materials and Equipments

Materials and equipments used in the present study are listed in Tables 2.1 and 2.2, respectively.

Table 2.1 Materials used in this study

Materials	Grade	Suppliers
Ortho unsaturated polyester 100%	ER-2595I 100%	Eternal Resin
Iso unsaturated polyester 100%	ER-2660 100%	Eternal Resin
Styrene monomer	Purum ≥ 99.0%(GC)	Unix Bright
Methyl methacrylate	Purum ≥ 99.0%(GC)	Bariki
Hydroquinone	Purum ≥ 99.0%	Eastman Chemical
Cobalt octoate 10%	Metal 10%	OMG Kokkola Chemical
Methyl ethyl ketone peroxide	Peroximon K10	Elf Atochem
Methanol	Commercial	Mtsubishi Gas Chemical, Singapore
Toluene	Commercial	Exxon chemical
KOH	AR	Merck
Tetrahydrofuran	AR	Merck
Glass fiber	450 g/m^2	Glass Fiber (JAPAN)

Table 2.2 Equipments used in this study

Equipments	Maker and model
Viscometer	Brookfield LVT DVII
Temperature Recorder	Lloyd Instrument Graphic 1002
Tensile and Bending Tester	Lloyd Instrument LR 50K
Hardness Tester	Matsuzawa DXT High Quality Rockwell
Accelerated Weathering Tester	The Panel Company (U.S.A.) QUV
Heat Distortion Temperature and Vicat Softening Point Temperature Tester	Toyo Seiki SN-F
Gel Permeation Chromatograph	Hewlett Package HP 1100

2.2 Preparation of Unsaturated Polyester Resin (UPR)

2.2.1 Orthophthalic Unsaturated Polyester Resin (Ortho UPR)

The preparation of ortho UPR is shown in Scheme 2.1. The 100% ortho UP was diluted with styrene monomer (SM) and methyl methacrylate (MMA) at different compositions listed in Table 2.3. Hydroquinone in methanol (125 ppm) was added as an inhibitor.

Scheme 2.1 Preparation of Ortho UPR

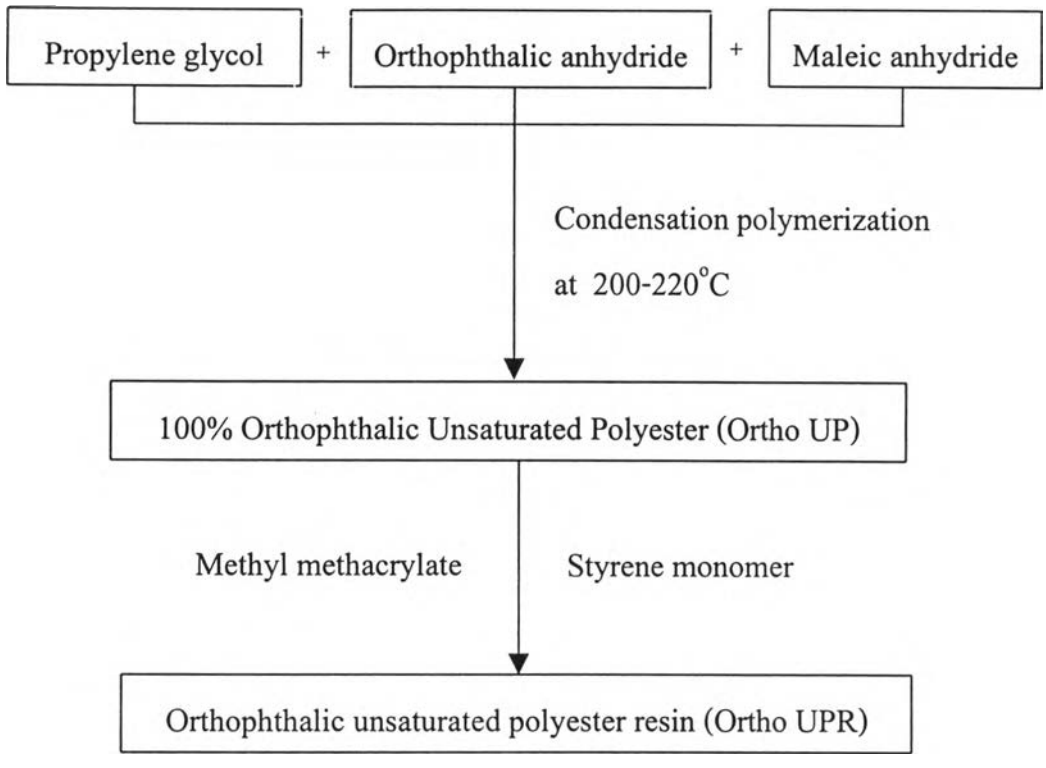


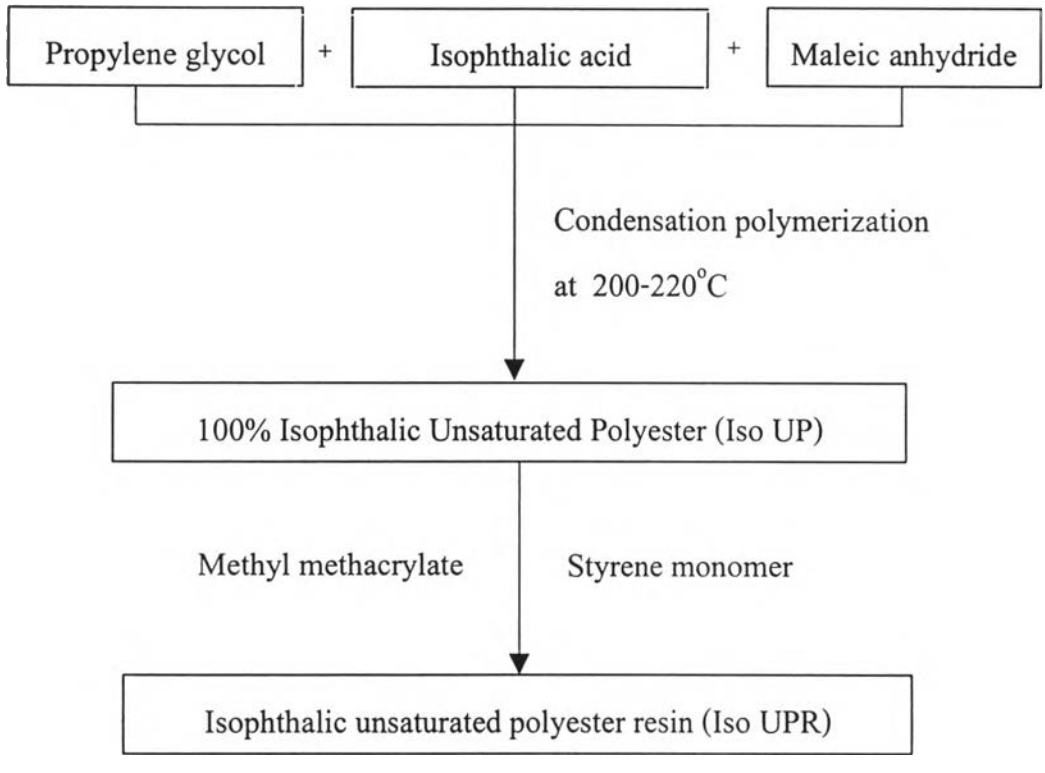
Table 2.3 Composition of starting materials in the ortho UPR formulation

Ortho UP (wt%)	SM (wt%)	MMA (wt%)
65	35	0
65	30	5
65	25	10
65	20	15
65	15	20
65	10	25
65	0	35

2.2.2 Isophthalic Unsaturated Polyester Resin (Iso UPR)

The preparation of iso UPR is shown in Scheme 2.2

Scheme 2.2 Preparation of iso UPR



The 100% iso UP was diluted with styrene monomer (SM) and methyl methacrylate (MMA) at different compositions listed in Table 2.4. Hydroquinone in methanol (125 ppm) was added as an inhibitor.

Table 2.4 Composition of starting materials in the iso UPR formulation

Iso UP (wt%)	SM (wt%)	MMA (wt%)
65	35	0
65	30	5
65	25	10
65	20	15
65	15	20
65	10	25
65	0	35

2.3 Characterization of UPR

2.3.1 Viscosity Determination

Viscosity of UPR samples were determined by using Brookfield LVT DVII viscometer with spindle number 3 at the speed of 60 rpm at 25°C.

2.3.2 Acid Value Determination

UPR sample (2-3 g) was dissolved in a mixture of toluene and methanol (70:30 v/v), then titrated with 0.1N KOH using phenolphthalein as an indicator. The method for calculation is described in Appendix A1.

2.3.3 Non-volatile Content Determination

UPR sample (1.2-1.3 g) was put into an aluminium foil cup and heated in an oven at 105°C for 3 hour. The method of calculation of non-volatile content is described in Appendix A2.

2.3.4 Gel Time Determination by using Cup Gel Test

UPR sample (50 g) was put into a plastic cup at 25°C. Cobalt octoate (0.2 wt% of UPR) was added and mixed together with a thermometer. While Methyl ethyl ketone peroxide (MEKPO) was added and stirred. Gel time is the time started until the mixture becomes a gel.

2.3.5 SPI Gel Test

UPR sample (50 g) was put into a plastic cup. Cobalt octoate (0.2 wt% of UPR) was added and mixed MEKPO (1.0 wt% of UPR) was then added. The mixture was put in a test tube in a 60°C waterbath. The temperature of the mixture was detected by using temperature recorder with type J thermocouple. The method of calculation of curing parameters is shown in Appendix A3.

2.3.6 Molecular Weight Determination

Dried UPR sample (0.02 g) UPR was dissolved in tetrahydrofuran (5 ml) then as investigated by gel permeation chromatograph to obtain the molecular weight.

2.4 Preparation of Thermoset Polyester (cured UPR)

Preparation the cured UPR was done by addition of cobalt octoate (0.2 wt% of UPR) followed by addition of MEKPO. The amounts of MEKPO used were 0.5, 1.0, 1.5 and 2.0% volume by weight (v/wt) of UPR.

The composition was mixed and degassed to remove air bubbles, then placed in a glass mould covered with Mylar film and separated by 2 cm rubber spacers. Samples were allowed to gel at room temperature for 24 h, followed by post curing in an oven at 100°C for 2 h.

Reinforced thermoset UPR was made with hand lay up by using chop-strand E 450 glass fiber.

2.5 Characterization of the Thermoset Polyester

(The details were described in Appendix B)

2.5.1 Tensile Strength

Method	:	ASTM D638
Equipment	:	Lloyd (50 KN Load cell)
Shape	:	dumbell
Dimension	:	13 mm width of narrow section 110 mm of gauge length and 165 mm of overall length.
Condition	:	test speed of 5 mm/min

Number of test	:	at least 5 specimens for each sample

2.5.2 Flexural Strength

Method	:	ASTM D790
Equipment	:	Lloyd (2.5 KN Load cell)
Shape	:	rectangular
Dimension	:	25 mm x 3 mm x 60 mm
Condition	:	support span of 48 mm and test speed of 1.2 mm/min
Number of test	:	at least 5 specimens for each sample

2.5.3 Heat DistortionTemperature

Method	:	ASTM D648
Equipment	:	Toyo seiki
Shape	:	rectangular
Dimension	:	13 mm x 3 mm x 127 mm
Number of test	:	at least 2 specimens for each sample

2.5.4 Hardness

Method	:	ASTM D785
Equipment	:	Matsuzawa DXT High quality Rockwell
Shape	:	rectangular
Dimension	:	7 mm thickness

Scales	:	100N load and 0.2500 in indenter diameter

2.5.5 Weathering Resistance (change in color)

Method	:	ASTM G53
Equipment	:	Q-U-V- accelerator weathering tester (The panel company)
Shape	:	rectangular
Dimension	:	3 mm thickness
Test condition	:	4 h UV at 60°C and 4 h condensation at 50°C