CHAPTER II

EXPERIMENTAL

2.1 Materials

Reagents and raw materials were obtained from the following suppliers:

1. **Allyl glycidyl ether :** Fluka

2. **1,4-Butanediol :** Thai Polyurethane Industry Co., Ltd.

3. **Benzoyl peroxide :** Fluka

4. **Bisphenol-A :** Fluka

5. **Methyl ethyl ketone peroxide :** Fluka

6. **Cobalt Naphthenate :** Sigma

7. **Dibutyltin dilaurate :** Fluka

8. **Polymeric MDI (Raypol C900) :** Thai Polyurethane Industry Co., Ltd.

9. **Polyester polyol (F 113) :** Thai Polyurethane Industry Co., Ltd.

10. **Styrene monomer :** Thai Polyurethane Industry Co., Ltd.

2.2 Equipments

1. **Nuclear Magnatic Resonance Spectrosocopy :** Bruker Model ACF200 Fourier-transform NMR Spectrometer
2. **Tensile Testing (ASTM D638) :** Instron Model 4301
3. **Dynamic Mechanical Analysis (DMA) :** Netzch DMA 242
4. **Thermogravimetric Analysis (TGA) :** Perkin Elmer TGA 7
5. **Scanning Electron Microscopy (SEM) :** JSM 35 CF

2.3 Preparation of Bis-(3-allyloxy-2-propanol) diphenylolpropane (BAPD)

BAPD was prepared according to the procedure described in the literature. A mixture of bisphenol-A (10.01 g, 0.044 mol), allyl glycidyl ether (10.05 g, 0.088 mol) and sodium hydroxide (3.62 g, 0.090 mol) was heated and stirred at 120°C for 3 hours, then taken up in ethyl acetate and washed with water. The organic layer was dried ($MgSO_4$) and concentrated under reduced pressure. The residue was purified by flash chromatography eluting with 5% ethyl acetate/hexane to give BAPD (18.20 g, 91 % based on bisphenol A) as a yellow liquid; ^{1}H NMR 7.13 (4H, d, J=8.8), 6.81 (4H, d, J=8.8), 5.82 (2H, tdd, J=5.7, 11.2, 7.2), 5.30 (2H, tdd, J=1.6, 3.2, 17.2), 5.16(2H, tdd, J=1.6, 3.3, 11.5), 4.09-4.18 (2H, m), 3 97-4.04 (8H, m), 3.61 (2H, dd, J=4.4, 9.6), 3.57 (2 H, dd, J=5.7, 9.6), 1.61 (6H, s).[26-27]

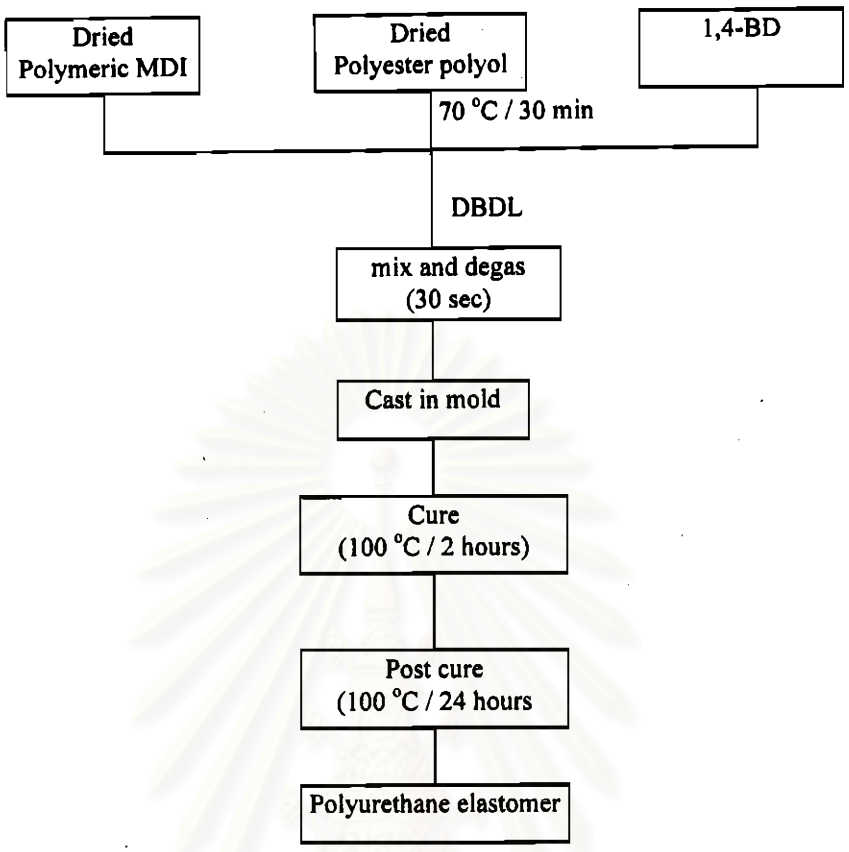
2.4 Preparation of Polyurethane Elastomers[2-3,6-18]

2.4.1 MDI:Polyol:1,4-BD Formulation

The NCO/OH ratio employed was constant at 1.04. Dibutyltin dilaurate (DBDL) was used as a catalyst (0.02% by weight of BAPD). 1,4-Butanediol (1,4-BD) was used as a chain extender. The equivalent weight ratio of MDI:Polyol:1,4-BD were varied as shown in Table 2.1. The experimental detail for the preparation of PU elastomer was shown in Scheme 2.1.

Table 2.1 Composition of starting materials in MDI:Polyol:1,4-BD formulation

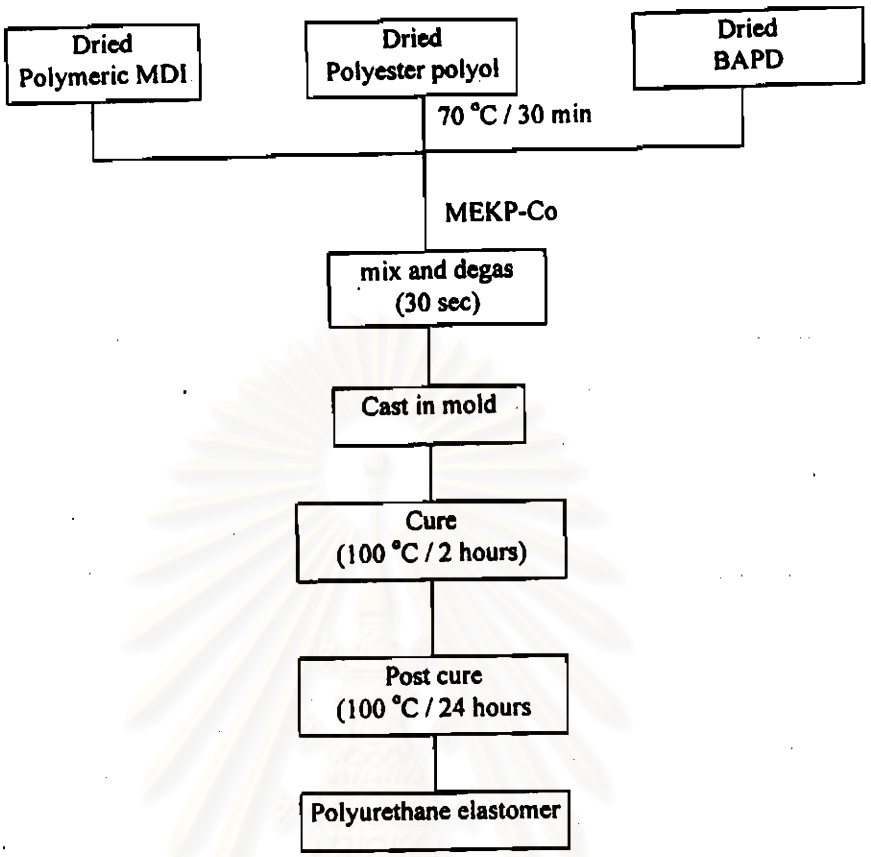
Equivalent weight ratio	**Weight of starting materials (g)**			
MDI:Polyol:1,4-BD	**MDI**	**Polyol**	**1,4-BD**	**DBDL**
2:1.8:0.2	14.316	52.726	2.368	0.014
2:1.4:0.6	20.923	46.236	4.846	0.014
2:1.0:1.0	11.333	58.438	1.125	0.014
2:0.6:1.4	9.067	60.107	0.301	0.015



Scheme 2.1 Procedure for the preparation of polyurethane elastomer from MDI:Polyol:1,4-BD formulation

2.4.2 MDI:Polyol:BAPD:MEKP-Co Formulation

The equivalent ratio of NCO/OH employed was constant at 1.04. Methyl ethyl ketone peroxide (MEKP) and cobalt naphthenate (Co) were used as initiator and accelerator, respectively. The amount of MEKP employed were 1.0, 2.0 and 5.0% by weight of BAPD. The content of Co was 25% of MEKP. DBDL was used as a catalyst (0.02% by weight of BAPD) The composition of MDI:Polyol:BAPD:MEKP-Co were varied as shown in Table 2.2. The experimental detail was shown in Scheme 2.2.



Scheme 2.2 Procedure for the preparation of polyurethane elastomer from MDI:Polyol:BAPD:MEKP-Co formulation

Table 2.2 Composition of starting materials in MDI:Polyol:BAPD:MEKP-Co formulation

Equivalent weight ratio MDI:Polyol:BAPD	Weight of starting materials (g)					
	MDI	Polyol	BAPD	DBDL	MEKP	Co
2:1.8:0.2	9.001	59.665	1.510	0.014	0.011[a]	0.004[a]
					0.023[b]	0.008[b]
					0.057[c]	0.019[c]
2:1.4:0.6	10.501	54.142	5.284	0.014	0.040[a]	0.013[a]
					0.079[b]	0.026[b]
					0.020[c]	0.066[c]
2:1.0:1.0	12.510	46.038	10.485	0.014	0.079[a]	0.026[a]
					0.157[b]	0.052[b]
					0.039[c]	0.131[c]
2:0.6:1.4	16.021	35.357	18.788	0.015	0.141[a]	0.047[a]
					0.282[b]	0.094[b]

[a] 1.0 wt% of BAPD

[b] 2.0 wt% of BAPD

[c] 5.0 wt% of BAPD

2.4.3 MDI:Polyol:BAPD:BP Formulation

The experiment was performed according to the procedure described in experiment 2.4.2 employing benzoyl peroxide (BP) as an initiator.

Table 2.3 Composition of starting materials in MDI:Polyol:BAPD:BP formulation

Equivalent weight ratio MDI:Polyol:BAPD	Weight of starting materials (g)				
	MDI	Polyol	BAPD	DBDL	BP
2:1.8:0.2	9.001	59.665	1.510	0.014	0.015[a] 0.030[b] 0.076[c]
2:1.4:0.6	10.501	54.142	5.284	0.014	0.053[a] 0.106[b] 0.264[c]
2:1.0:1.0	12.510	46.038	10.485	0.014	0.105[a] 0.210[b] 0.524[c]
2:0.6:1.4	16.021	35.357	18.788	0.015	0.188[a] 0.376[b]

[a] 1.0 wt% of BAPD

[b] 2.0 wt% of BAPD

[c] 5.0 wt% of BAPD

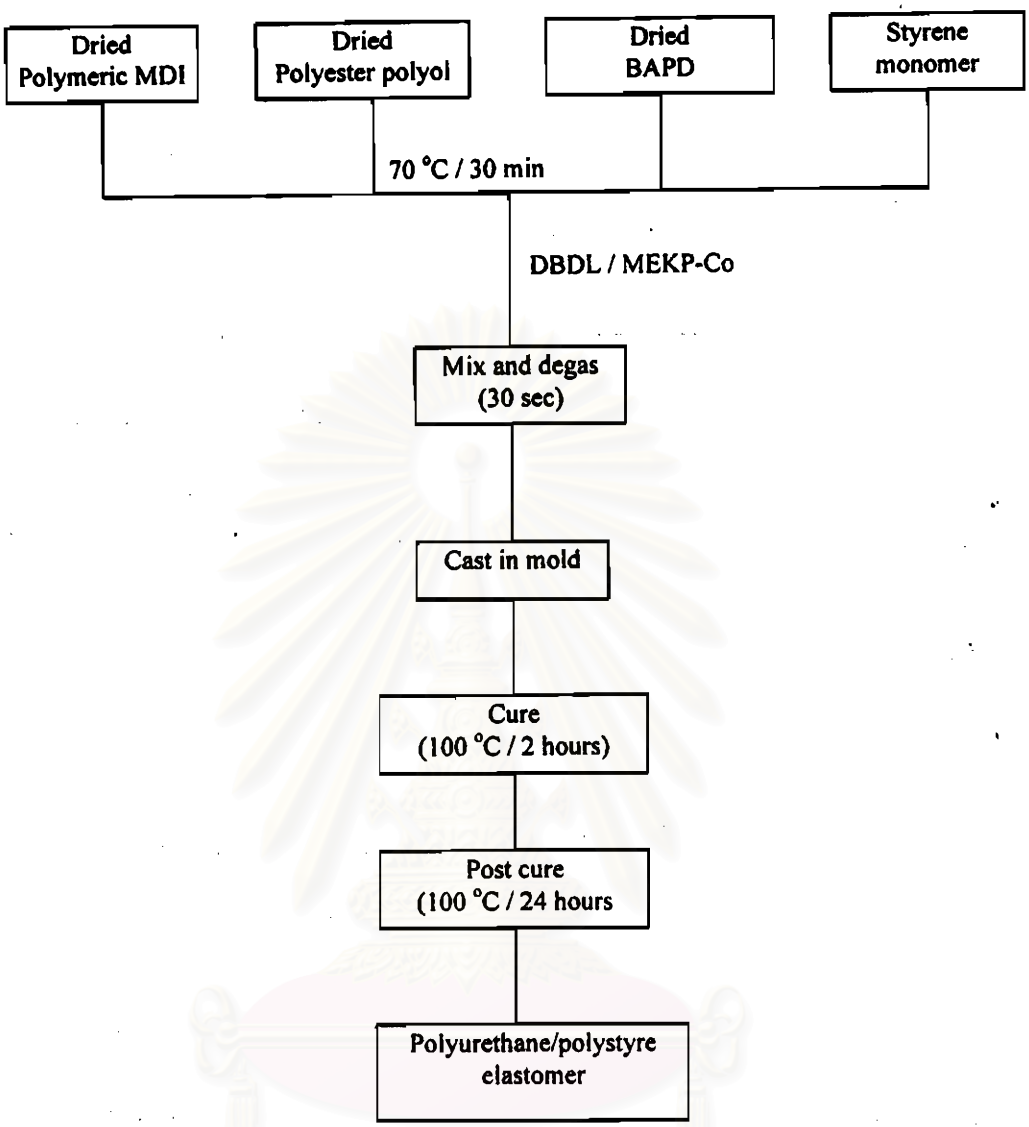
2.5 Preparation of Polyurethane/Polystyrene Elastomer [2-3,6-18]

2.5.1 MDI:Polyol:BAPD:Styrene:MEKP-Co Formulation

The experiment was performed according to the procedure described in experiment 2.4.2 and styrene monomer was added to the formulation as shown in Table 2.4. The procedure for the preparation of PU/PS elastomer was shown in Scheme 2.3.

Table 2.4 Composition of starting materials in MDI:Polyol: BAPD:Styrene:MEKP-Co formulation

Equivalent weight ratio MDI:POLYOL:BAPD	% Styrene monomer (wt%)	Weight of Styrene monomer (g)
2:1.8:0.2	5	3.627
	10	7.097
	15	10.529
	20	14.038
2:1.4:0.6	5	3.768
	10	7.273
	15	10.505
	20	14.006
2:1.0:1.0	5	3.739
	10	7.200
	15	10.385
	20	13.846



Scheme 2.3 Procedure for the preparation of polyurethane/ polystyrene elastomers.

2.6 Mechanical Testing

Mechanical Testing of the PU and PU/PS elastomers were performed according to the procedure described in ASTM D638 (tensile testing).