

CHAPTER II

EXPERIMENTS

2.1 Materials

All reagents and solvents were of analytical grade quality. All chemicals were obtained from Baker, Fluka and Aldrich and were used as received. Zinc (II) acetate dihydrate, nickel (II) acetate tetrahydrate, manganese (II) acetate tetrahydrate, cobalt (II) acetate tetrahydrate, 2,4-dihydroxybenzaldehyde, triethylenetetramine, tolylene 2,4-diisocyanate terminated poly (1,4-butanediol) prepolymer (PB900), MW 900 g/mol and tolylene 2,4-diisocyanate terminated poly (propylene glycol) prepolymer (PP1000), MW 1000 g/mol were used without further purification.

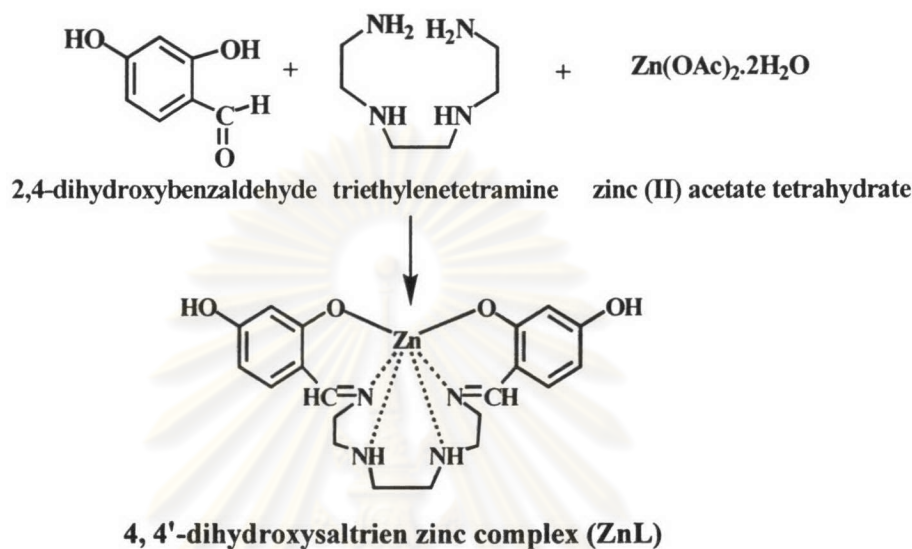
2.2 Analytical procedures

IR spectra of the samples were recorded on a Nicolet Impact 410 using KBr pellet method. Elemental analyses were carried out on a Perkin Elmer Elemental Analyzer 2400 CHN. Matrix-assisted laser desorption ionization-time of flight (MALDI-TOF) mass spectra were obtained on a Bruker Biflex mass spectrometer by using α -cyanocinnamic acid as a matrix. NMR spectra were recorded in CDCl_3 and $\text{DMSO}-d_6$ solution on a Varian Mercury-400 BB instrument. Chemical shifts are given in parts per million (ppm) using the proton residual as internal reference. The thermal properties were studied using a Perkin-Elmer DSC 7 differential scanning calorimeter. All the samples were heated in DSC cell using closed aluminium pan under air with the heating rate of 20 °C/min. Thermogravimetric measurements were performed on a Netzsch thermogravimetric analyzer (STA 409C) at the heating rate 20 °C/min under air/nitrogen (50/50) atmosphere. Initial decomposition temperature (IDT) was reported at the temperature where weight loss of the samples was observed. Thermal stability of the polymers were determined using isothermal TGA by heating the polymer samples in a heated air oven at 170 °C for 8 h and their weight losses were then measured. Solubility of the polymers was tested in various polar and nonpolar solvents by adding of 10 mg samples to 2 ml of a solvent.

2.3 Synthetic procedures

2.3.1 Synthesis of hexadentate Schiff base metal complexes (ML)

2.3.1.1 Synthesis of 4,4'-dihydroxysaltrien zinc complex (ZnL)



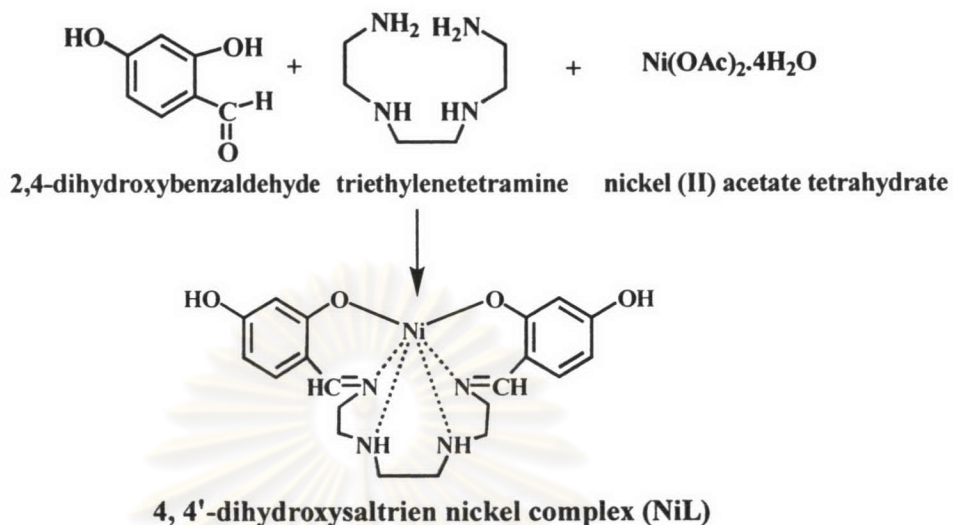
The synthesis of ZnL was carried out according to the method reported in the literature[2]. The mixture of 2, 4-dihydroxybenzaldehyde (0.463 g, 3.35 mmol,) and zinc (II) acetate dihydrate (0.365 g, 1.67 mmol) in methanol (20 ml) was prepared, then cooled to 0 °C. A cool (0 °C) methanolic solution (10 ml) of triethylenetetramine was added dropwise over a period of 10 minutes. The mixture was stirred for 15 minutes and neutralized by adding a solution of 2M sodium hydroxide solution (1.67 mL, 1.67 mmol) and stirred at 0 °C for 30 minutes. The pink powder precipitated immediately when adding solution of sodium hydroxide and was isolated by filtration, then the yellow solution of filtrate was allowed to stand at room temperature for 6 hours. The yellow crystal (ZnL) formed was filtered off and washed with methanol and acetone. Both the pink powder and ZnL were dried *in vacuo* to remove traces of the solvent. Table 2.1 shows the composition of starting materials that varied the solution of base and mole ratio of 2, 4-dihydroxybenzaldehyde: zinc (II) acetate dihydrate: triethylenetetramine. From the Table 2.1 indicates that the best yield of ZnL (51%) was obtained when the mole ratio of 2, 4-dihydroxybenzaldehyde: zinc (II) acetate dihydrate: triethylenetetramine was taken as 2: 1: 1 and solution of base was potassium carbonate.

Table 2.1 Composition of starting materials in the synthesis of 4,4'-dihydroxysaltrien zinc complex (ZnL)

Mole ratio of of 2, 4-dihydroxybenzaldehyde: zinc (II) acetate dihydrate: triethylenetetramine	Volume of triethylenetetramine (mL)	Concentration, Volume of base (mL)	Yield of the pink powder (%)	Yield of ZnL (%)
2 : 1 : 1	0.25 mL, 1.67 mmol	NaOH 2M, 1.67 mL	16	13
2 : 1 : 1	0.25 mL, 1.67 mmol	NaHCO ₃ 2M, 1.67 mL	11	9
2 : 1 : 1	0.25 mL, 1.67 mmol	Na ₂ CO ₃ 1M, 1.67 mL	14	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	KOH 1M, 1.67 mL	13	27
2 : 1 : 1	0.25 mL, 1.67 mmol	K ₂ CO ₃ 1M, 1.67 mL	10	51
2 : 1 : 2	0.50 mL, 3.34 mmol	K ₂ CO ₃ 1M, 1.67 mL	N/A	11

ZnL: IR (KBr, cm⁻¹); 3315 (NH), 2857, 1629 (C=N), 1601, 1449, 1378, 1279, 1216, 1118, 1026, 980, 893, 841, 790, 661, 558. ¹H NMR (400 MHz, DMSO-*d*₆+CDCl₃, ppm); δ 9.06 (s, 2H, OH), 8.10 (s, 2H, CH=N), 6.86-6.84 (d, 2H, ArH, *J*= 8.18 Hz), 5.83-5.81 (d, 2H, ArH, *J*= 7.78 Hz), 5.77 (s, 2H, ArH). 3.70-3.66 (t, 2H, CH₂, *J*= 13.03 Hz), 3.14-3.13 (m, 2H, CH₂), 2.94 (s, 2H, CH₂), 2.81-2.80 (d, 2H, CH₂, *J*= 6.48 Hz), 2.54-2.53 (m, 2H, CH₂), 2.41-2.39 (d, 2H, CH₂, *J*= 6.88 Hz). MALDI-TOF MS (*m/z*) 450.1 (C₂₀H₂₄O₄N₄Zn). Anal. Calcd. For C₂₀H₂₄O₄N₄Zn.3H₂O: C 47.67; H 6.00; N 11.12; found C; 48.25, H; 5.93, N; 11.62.

2.3.1.2 Synthesis of 4, 4'-dihydroxysaltrien nickel complex (NiL)



The experiment was performed according to the procedure described in experiment 2.3.1.1 employing nickel (II) acetate tetrahydrate (0.415 g, 1.67 mmol) instead of zinc (II) acetate dihydrate (0.365 g, 1.67 mmol). The pink powder precipitated from the solution immediately then it was isolated by filtration. The nickel complex (NiL) which was a green crystal crystallized when the filtrate was stood at room temperature for 6 hours. The pink powder and NiL were washed with methanol and acetone several times and then were dried *in vacuo*. Table 2.2 shows the composition of starting materials that varied the solution of base and mole ratio of 2,4-dihydroxybenzaldehyde: nickel (II) acetate tetrahydrate: triethylenetetramine. The best yield of NiL (89%) was obtained when the mole ratio of 2,4-dihydroxybenzaldehyde: nickel (II) acetate tetrahydrate: triethylenetetramine was taken as 2: 1: 2 and solution of 1 M of potassium carbonate was used as base.

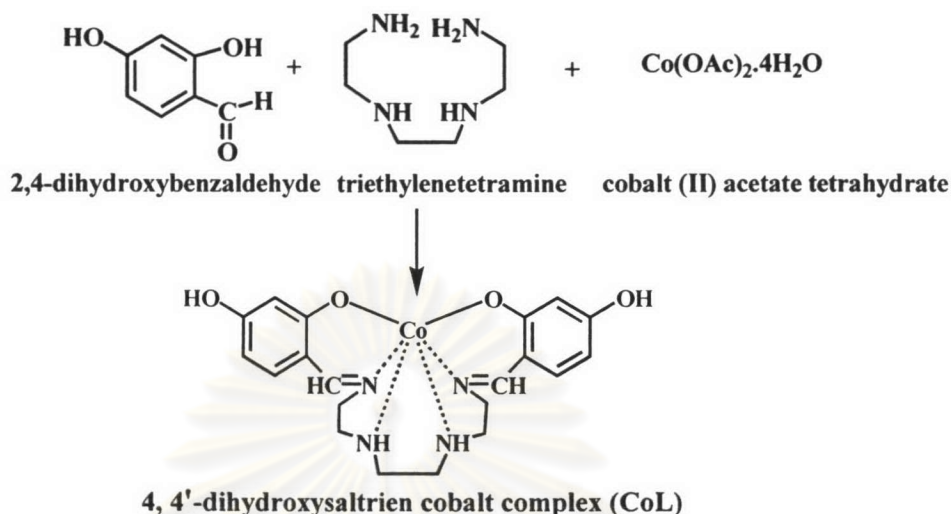
Table 2.2 Composition of starting materials in the synthesis of 4, 4'-dihydroxysaltrien nickel complex (NiL)

Mole ratio of of 2, 4-dihydroxybenzaldehyde: nickel (II) acetate tetrahydrate: triethylenetetramine	Volume of triethylenetetramine (mL)	Concentration, Volume of base (mL)	Yield of the pink powder (%)	Yield of NiL (%)
2 : 1 : 1	0.25 mL, 1.67 mmol	NaOH 2M, 1.67 mL	14	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	K ₂ CO ₃ 1M, 1.67 mL	12	24
2 : 1 : 2	0.50 mL, 3.34 mmol	NaOH 2M, 1.67 mL	N/A	57
2 : 1 : 2	0.50 mL, 3.34 mmol	NaHCO ₃ 2M, 1.67 mL	N/A	74
2 : 1 : 2	0.50 mL, 3.34 mmol	Na ₂ CO ₃ 1M, 1.67 mL	N/A	78
2 : 1 : 2	0.50 mL, 3.34 mmol	KOH 2M, 1.67 mL	N/A	80
2 : 1 : 2	0.50 mL, 3.34 mmol	K ₂ CO ₃ 1M, 1.67 mL	N/A	89

NiL: IR (KBr, cm⁻¹); 3312 (NH), 2868, 1634 (C=N), 1598, 1449, 1374, 1335, 1279, 1214, 1118, 1030, 989, 907, 841, 789, 661, 559. MALDI-TOF MS (*m/z*) 443.5 (C₂₀H₂₄O₄N₄Ni). Anal. Calcd. For C₂₀H₂₄O₄N₄Ni.3H₂O: C 48.32; H 6.08; N 11.27; Found C 48.26; H 6.22; N 11.55.

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2.3.1.3 Synthesis of 4, 4'-dihydroxysaltrien cobalt complex (CoL)



The experiment was performed according to the procedure described in experiment 2.3.1.1 employing cobalt (II) acetate tetrahydrate (0.416 g, 1.67 mmol) instead of zinc (II) acetate dihydrate (0.0365 g, 1.67 mmol). The brown powder precipitated from the solution immediately then it was filtrated. The cobalt complex (CoL) which was a dark-brown powder precipitated when the filtrate was stood at room temperature for 6 hours. The brown and dark-brown powders were washed with methanol and acetone several times and then were dried *in vacuo*. Table 2.3 shows the composition of starting materials that varied the solution of base and mole ratio of 2,4-dihydroxybenzaldehyde: cobalt (II) acetate tetrahydrate: triethylenetetramine. The best yield of CoL complex (31%) was obtained when the mole ratio of 2,4-dihydroxybenzaldehyde: cobalt (II) acetate tetrahydrate: triethylenetetramine was taken as 2: 1: 1 and 1 M potassium carbonate was used as base.

However, the CoL complex decomposed when was dried. The color of solid changed from dark brown to black. CoL complex was unstable as a solid, so the polymerization reaction of CoL complex and prepolymer was not synthesized.

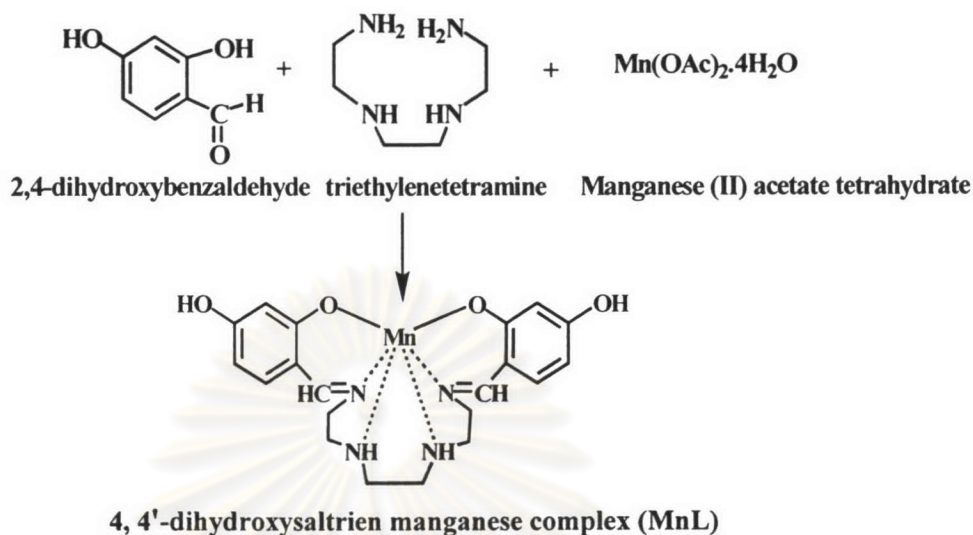
Table 2.3 Composition of starting materials in the preparation of 4, 4'-dihydroxysaltrien cobalt complex (CoL)

Mole ratio of of 2, 4-dihydroxybenzaldehyde: cobalt (II) acetate tetrahydrate: triethylenetetramine	Volume of triethylenetetramine (mL)	Concentration, Volume of base (mL)	Yield of the brown powder (%)	Yield of CoL (%)
2 : 1 : 1	0.25 mL, 1.67 mmol	NaOH 2M, 1.67 mL	28	26
2 : 1 : 1	0.25 mL, 1.67 mmol	NaHCO ₃ 2M, 1.67 mL	31	28
2 : 1 : 1	0.25 mL, 1.67 mmol	Na ₂ CO ₃ 1M, 1.67 mL	28	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	KOH 1M, 1.67 mL	N/A	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	K ₂ CO ₃ 1M, 1.67 mL	32	31
2 : 1 : 2	0.50 mL, 3.34 mmol	K ₂ CO ₃ 1M, 1.67 mL	N/A	N/A
2 : 1 : 2	0.50 mL, 3.34 mmol	-	N/A	N/A
2 : 1 : 3	0.75 mL, 4.01 mmol	-	N/A	N/A

CoL: IR (KBr, cm⁻¹); 3387 (NH), 2933, 1634 (C=N), 1590, 1538, 1449, 1358, 1226, 1125, 804, 472.

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2.3.1.4 Synthesis of 4, 4'-dihydroxysaltrien manganese complex (MnL)



The experiment was performed according to the procedure described in experiment 2.3.1.1 employing manganese (II) acetate tetrahydrate (0.410 g, 1.67 mmol) instead of zinc (II) acetate dihydrate (0.0365 g, 1.67 mmol). The brown powder precipitated from the solution immediately then it was filtrated off and then the brown solution of filtrate was allowed to stand at room temperature for 6 hours. The dark-brown powder (MnL) precipitated and was filtered off and then was washed with methanol and acetone. The brown powder and MnL were dried *in vacuo*. The solution of base and mole ratio of 2,4-dihydroxybenzaldehyde: manganese (II) acetate tetrahydrate: triethylenetetramine was varied as shown in Table 3.4. The best yield of MnL complex (81%) was obtained when the mole ratio of 2,4-dihydroxybenzaldehyde: manganese (II) acetate dihydrate: triethylenetetramine was taken as 2: 1: 1 without the neutralization with base.

However, the MnL complex decomposed when was dissolved in solvent. The color of dissolved solution changed from dark brown to black. Therefore, the polymerization reaction of MnL complex and prepolymer was not performed.

Table 2.4 Composition of starting materials in the preparation of 4,4'- dihydroxysaltrien manganese complex (MnL)

Mole ratio of of 2, 4-dihydroxybenzaldehyde: cobalt (II) acetate tetrahydrate: triethylenetetramine	Volume of triethylenetetramine (mL)	Concentration, Volume of base (mL)	Yield of the brown powder (%)	Yield of MnL (%)
2 : 1 : 1	0.25 mL, 1.67 mmol	NaOH 2M, 1.67 mL	25	27
2 : 1 : 1	0.25 mL, 1.67 mmol	NaHCO ₃ 2M, 1.67 mL	17	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	Na ₂ CO ₃ 1M, 1.67 mL	8	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	KOH 1M, 1.67 mL	N/A	N/A
2 : 1 : 1	0.25 mL, 1.67 mmol	K ₂ CO ₃ 1M, 1.67 mL	23	22
2 : 1 : 2	0.50 mL, 3.34 mmol	K ₂ CO ₃ 1M, 1.67 mL	N/A	N/A
2 : 1 : 2	0.50 mL, 3.34 mmol	-	N/A	81

MnL: IR (KBr, cm⁻¹); 3421 (NH), 2924, 1581 (C=N), 1481, 1357, 1300, 1232, 1183, 1123, 980, 850, 641. Anal. Calcd. For C₂₀H₂₄O₄N₄Mn.H₂O: C 52.52; H 5.73; N 12.25; Found C 52.30; H 5.65; N 12.63.

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2.3.1 Investigation of the polymerization reaction of polyurethane-ureas and copolyurethane-ureas

2.3.1.1 Differential Scanning Calorimetry (DSC) of polymerization reaction of PB900 and ZnL

ZnL and PB900 were mixed at the mole ratio of ZnL: PB900 = 1: 1. The sample was then heated in DSC cell using a closed aluminium pan under nitrogen. The temperature range was 25-300 °C at heating rate of 20 °C/min.

2.3.1.2 Infrared Spectroscopy (IR) of polymerization reaction of prepolymer and ML

Metal complexes, diisocyanates and xylylenediamine were mixed and was then heated in a hot air oven at 120 °C. IR spectroscopy was used to follow the progress of the reaction. The completeness of polymerization was confirmed by the disappearance of the characteristic band of the isocyanate groups at 2275 cm^{-1} . Weight ratios of metal complex: diisocyanate were varied as shown in Table 2.5. In the polymer codes, codes of the synthesized are abbreviated as M-P-D-X. M represents the metal complex (Zn = ZnL, Ni = ZnL), P indicates the type of prepolymer (PB = PB900 and PP = PP1000), D refers to xylylenediamine and X refers to the mole ratio of the M: P: D.

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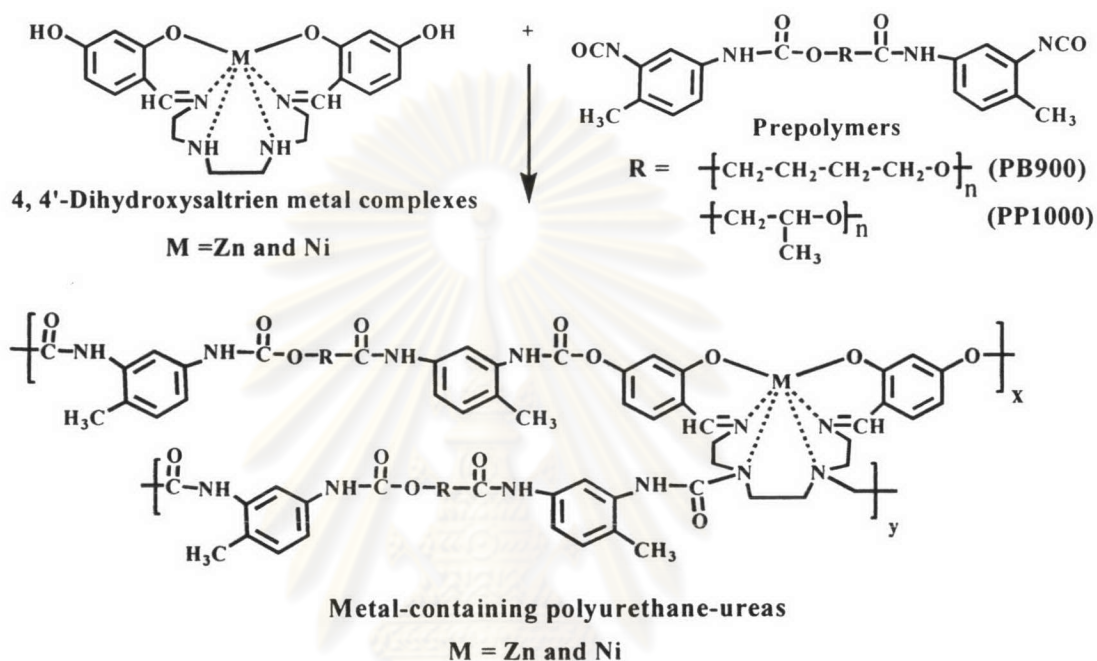
Table 2.5 Composition of starting materials of the polymerization reaction between ML, xylylenediamine and prepolymer

Polymer Code	Weight (g), mole of starting materials (mole)					Reaction time (h)
	ZnL	NiL	PB900	PP1000	Xylylenediamine	
Zn-PB-11	0.041 g, 8.14x10 ⁻⁵ mol	-	0.073 g, 8.14x10 ⁻⁵ mol	-	-	4
Zn-PB-12	0.039 g, 7.74x10 ⁻⁵ mol	-	0.139 g, 1.54x10 ⁻⁴ mol	-	-	4
Zn-PB-13	0.039 g, 7.74x10 ⁻⁵ mol	-	0.209 g, 2.32x10 ⁻⁴ mol	-	-	^a
Zn-PP-12	0.032 g, 6.35x10 ⁻⁵ mol	-	-	0.127g, 1.27x10 ⁻⁴ mol	-	5
Ni-PB-11	-	0.109 g, 2.19x10 ⁻⁴ mol	0.197 g, 2.19x10 ⁻⁴ mol	-	-	3
Ni-PB-12	-	0.041 g, 8.26x10 ⁻⁵ mol	0.148 g, 1.65x10 ⁻⁴ mol	-	-	2
Ni-PB-13	-	0.027 g, 5.43x10 ⁻⁵ mol	0.147 g, 1.63x10 ⁻⁴ mol	-	-	^a
Ni-PP-12	-	0.033 g, 6.64x10 ⁻⁵ mol	-	0.133 g, 1.33x10 ⁻⁵ mol	-	4
Zn-PB-D-131	0.100 g, 1.98x10 ⁻⁴ mol	-	0.540 g, 6.00x10 ⁻⁴ mol	-	0.027 g, 1.98x10 ⁻⁴ mol	3
Zn-PP-D-131	0.074 g, 1.47x10 ⁻⁴ mol	-	-	4.440 g, 4.40x10 ⁻⁴ mol	0.020 g, 1.47x10 ⁻⁴ mol	3
Ni-PB-D-131	-	0.099 g, 1.99x10 ⁻⁴ mol	0.540 g, 6.00x10 ⁻⁴ mol	-	0.027 g, 1.98x10 ⁻⁴ mol	3
Ni-PP-D-131	-	0.099 g, 2.00x10 ⁻⁴ mol	-	0.600 g, 6.00x10 ⁻⁴ mol	0.027 g, 1.98x10 ⁻⁴ mol	3
PB-D-11	-	-	0.500 g, 5.55x10 ⁻⁴ mol	-	0.056 g, 5.51x10 ⁻⁴ mol	4
PP-D-11	-	-	-	0.500 g, 5.00x10 ⁻⁴ mol	0.068 g, 4.99x10 ⁻⁴ mol	4

^aAlthough polymerization was done for 8 hours, the –NCO peak of PB900 was still observed. This indicates that part of PB900 did not undergo polymerization reaction.

2.3.3 Synthesis of metal-containing polyurethane-ureas and copolyurethane-ureas

2.3.3.1 Synthesis of metal-containing polyurethane-ureas from ML and prepolymer



A mixture of the metal complex and prepolymer was heated in a hot air oven at 120 °C for 24 hours. The reaction mixture was dissolved in 3.0 ml of DMSO at 90 °C and was then precipitated in 10.0 ml of distilled water. The resulting elastomer was filtered off and was dried under vacuum to give brown elastomer. The weight ratios of the metal complex: prepolymer as shown in Table 2.6.

Table 2.6 Composition of starting materials in the synthesis of metal-containing polyurethanes-ureas

Polymer Code	Weight (g), mole of starting materials (mole)				Yield (%)
	ZnL	NiL	PB	PP	
Zn-PB-11	0.041 g, 8.13x10 ⁻⁵ mol	-	0.073 g, 8.11x10 ⁻⁵ mol	-	40
Zn-PB-12	0.039 g, 7.74x10 ⁻⁵ mol	-	0.139 g, 1.54x10 ⁻⁴ mol	-	79
Zn-PB-13	0.039 g, 7.74x10 ⁻⁵ mol	-	0.209 g, 2.32x10 ⁻⁴ mol	-	80
Ni-PB-11	-	0.109 g, 2.19x10 ⁻⁴ mol	0.197 g, 2.19x10 ⁻⁴ mol	-	41
Ni-PB-12	-	0.041 g, 8.24x10 ⁻⁵ mol	0.148 g, 1.64x10 ⁻⁴ mol	-	76
Ni-PB-13	-	0.028 g, 5.43x10 ⁻⁵ mol	0.148 g, 1.64x10 ⁻⁴ mol	-	80
Zn-PP-12	0.032 g, 6.35x10 ⁻⁵ mol	-	-	0.127 g, 1.27x10 ⁻⁴ mol	82
Ni-PP-12	-	0.033 g, 6.64x10 ⁻⁵ mol	-	0.132 g, 1.32x10 ⁻⁴ mol	83

Zn-PB-11: IR (KBr, cm⁻¹); 3346 (NH), 2940, 2860, 2798, 1716 (C=O), 1625 (C=N), 1608, 1541, 1479, 1448, 1370, 1224, 1105, 1069, 999, 885, 815, 768

Zn-PB-12: IR (KBr, cm⁻¹); 3304 (NH), 2935, 2860, 2796, 1725 (C=O), 1634 (C=N), 1600, 1559, 1539, 1480, 1450, 1371, 1222, 1113, 884

Zn-PB-13: IR (KBr, cm⁻¹); 3311 (NH), 2940, 2860, 2799, 1713 (C=O), 1606 (C=N), 1540, 1448, 1371, 1225, 1107, 877

Ni-PB-11: IR (KBr, cm⁻¹); 3387 (NH), 2940, 2859, 2791, 1718 (C=O), 1607 (C=N), 1542, 1448, 1370, 1226, 1107, 881

Ni-PB-12: IR (KBr, cm⁻¹); 3400 (NH), 2932, 2858, 2793, 1718 (C=O), 1606 (C=N), 1542, 1448, 1406, 1370, 1226, 1107, 879

Ni-PB-13: IR (KBr, cm⁻¹); 3397 (NH), 2932, 2858, 2796, 1719 (C=O), 1606 (C=N), 1542, 1448, 1225, 1107, 879

Zn-PP-12: IR (KBr, cm⁻¹); 3382 (NH), 2974, 2930, 2865, 1726 (C=O), 1634 (C=N), 1600, 1541, 1452, 1374, 1225, 1106, 925, 852

Ni-PP-12: IR (KBr, cm^{-1}); 3447 (NH), 2974, 2930, 2817, 1720 (C=O), 1617 (C=N), 1540, 1452, 1368, 1229, 1099, 1011, 933, 867, 817, 770

2.3.3.2 Synthesis of metal-containing copolyurethane-ureas from ML, prepolymers and xylylenediamines

The experiment was performed according to the procedure described in experiment 2.3.3.1. All starting materials were mixed and were then heated in a hot air oven at 120 °C for 24 hours. The reaction mixture was dissolved in 3.0 ml of DMSO at 90 °C and was then precipitated in 10.0 ml of distilled water. The resulting elastomer was filtered off and was dried under vacuum. The weight ratios of metal complex: prepolymer: xylylenediamine as shown in Table 2.7.

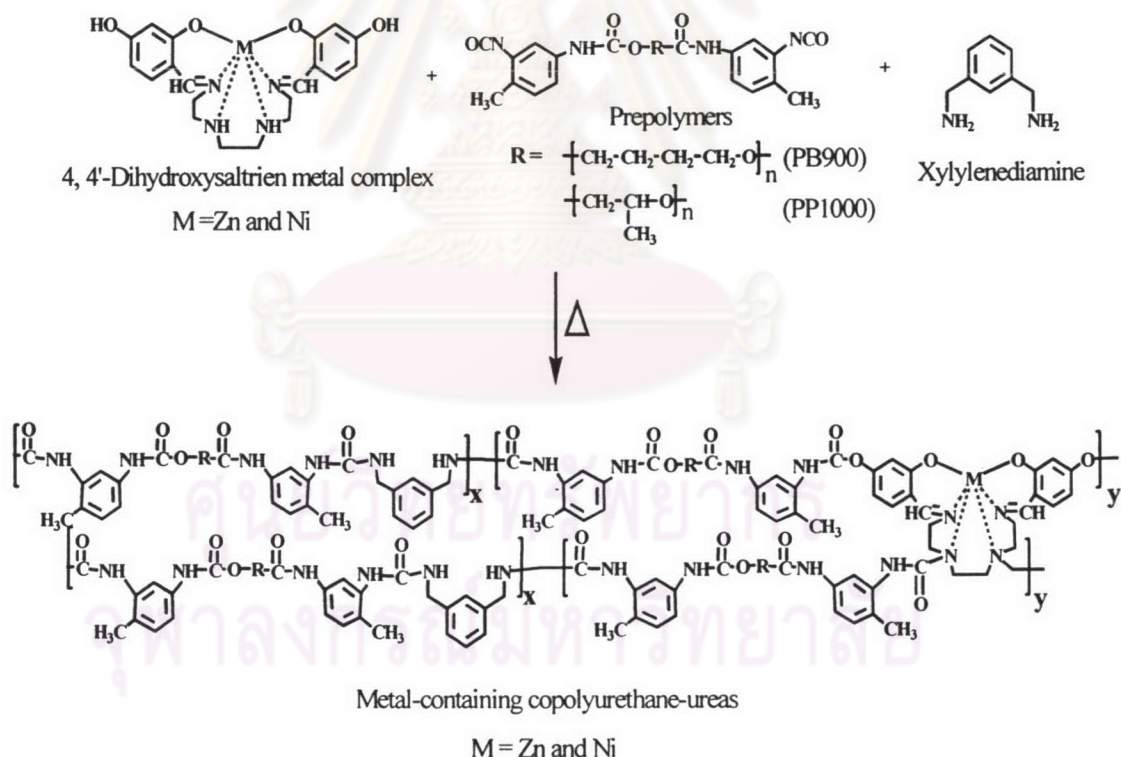


Table 2.7 Composition of starting materials in the synthesis of metal-containing copolyurethanes-ureas

Polymer Codes	Weight (g), mole of starting materials (mole)					Yield (%)
	ZnL	NiL	PB	PP	D	
Zn-PB-D-153	0.044 g, 8.73x10 ⁻⁵ mol	-	0.395 g, 4.39x10 ⁻⁴ mol	-	0.036 g, 2.64x10 ⁻⁴ mol	65
Zn-PB-D-131	0.100 g, 1.98x10 ⁻⁴ mol	-	0.535 g, 5.94x10 ⁻⁴ mol	-	0.027 g, 1.98x10 ⁻⁴ mol	70
Zn-PB-D-371	0.096 g, 1.90x10 ⁻⁴ mol	-	0.401 g, 4.46x10 ⁻⁴ mol	-	0.009 g, 6.61x10 ⁻⁵ mol	73
Ni-PB-D-153	-	0.044 g, 8.85x10 ⁻⁵ mol	0.397 g, 4.41x10 ⁻⁴ mol	-	0.036 g, 2.64x10 ⁻⁴ mol	64
Ni-PB-D-131	-	0.099 g, 1.99x10 ⁻⁴ mol	0.536 g, 5.96x10 ⁻⁴ mol	-	0.027 g, 1.98x10 ⁻⁴ mol	65
Ni-PB-D-371	-	0.094 g, 1.89x10 ⁻⁴ mol	0.400 g, 4.44x10 ⁻⁴ mol	-	0.009 g, 6.61x10 ⁻⁵ mol	68
Zn-PP-D-131	0.074 g, 1.47x10 ⁻⁴ mol	-	-	4.440 g, 4.40x10 ⁻⁴ mol	0.020 g, 1.47x10 ⁻⁴ mol	85
Ni-PP-D-131	-	0.099 g, 2.00x10 ⁻⁴ mol	-	0.600 g, 6.00x10 ⁻⁴ mol	0.027 g, 1.98x10 ⁻⁴ mol	83
PB-D-11	-	-	0.500 g, 5.55x10 ⁻⁴ mol	-	0.056 g, 5.51x10 ⁻⁴ mol	56
PP-D-11	-	-	-	0.500 g, 5.00x10 ⁻⁴ mol	0.068 g, 4.99x10 ⁻⁴ mol	60

Zn-PB-D-131: IR (KBr, cm⁻¹); 3363 (NH), 2939, 2860, 2796, 1711 (C=O), 1655 (C=O(C=N)), 1604, 1543, 1448, 1375, 1226, 1106, 879, 805, 765

Zn-PB-D-153: IR (KBr, cm⁻¹); 3363 (NH), 2938, 2870, 2796, 1712 (C=O), 1650 (C=O(C=N)), 1604, 1543, 1448, 1375, 1226, 1106, 879, 808, 765

Zn-PB-D-371: IR (KBr, cm⁻¹); 3363 (NH), 2939, 2863, 2799, 1716 (C=O), 1655 (C=O(C=N)), 1606, 1543, 1448, 1375, 1226, 1106, 879, 803, 769

Ni-PB-D-131: IR (KBr, cm⁻¹); 3344 (NH), 2938, 2859, 2795, 1711 (C=O), 1662 (C=O)(C=N), 1603, 1542, 1483, 1448, 1370, 1227, 1107, 878, 817, 770

Ni-PB-D-153: IR (KBr, cm⁻¹); 3344 (NH), 2938, 2859, 2795, 1711 (C=O), 1668 (C=O)(C=N), 1602, 1542, 1483, 1447, 1370, 1223, 1104, 878, 817, 773

Ni-PB-D-371: IR (KBr, cm⁻¹); 3344 (NH), 2939, 2855, 2794, 1710 (C=O), 1664 (C=O)(C=N), 1605, 1542, 1483, 1448, 1370, 1227, 1103, 878, 817, 774

Zn-PP-D-131: IR (KBr, cm^{-1}); 3312 (NH), 2973, 2924, 2871, 1727 (C=O), 1638 (C=N)(C=O), 1599, 1541, 1452, 1415, 1374, 1224, 1108, 924, 853, 773

Ni-PP-D-131: IR (KBr, cm^{-1}); 3316 (NH), 2973, 2924, 2871, 1718 (C=O), 1658 (C=O (C=N), 1603, 1542, 1451, 1376, 1229, 1101, 928, 866, 824, 765

PB-D-11: IR (KBr, cm^{-1}); 3341 (NH), 2973, 2932, 2871, 1717 (C=O), 1651 (C=O), 1601, 1547, 1451, 1378, 1231, 1101, 928, 874, 812, 773, 699, 661

PP-D-11: IR (KBr, cm^{-1}); 3344 (NH), 2938, 2859, 2800, 1711 (C=O), 1650 (C=O), 1603, 1542, 1480, 1448, 1370, 1227, 1107, 813, 766, 708, 669



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