

CHAPTER IV

RESULTS AND DISCUSSION

4.1 The Effects of Solvents

The influence of solvent types was investigated by propylene polymerization using two different catalyst systems, supported Ziegler-Natta catalyst ($\text{MgCl}_2/\text{TiCl}_4/\text{DEP-TEA}$) and in-situ supported metallocene catalyst ($\text{SiO}_2/\text{MAO}/\text{TMA}/\text{rac-Et}(\text{Ind})_2\text{ZrCl}_2$). Series of propylene polymerizations were performed with various types of solvent such as hexane, toluene, heptane and xylene.

4.1.1 The Effect of Solvents on The Catalytic Activity

The polymerizations were performed and the results are shown in Table 4.1.

Table 4.1 Catalytic activity using different types of solvent ^{a)}

Catalyst ^{b)}	Solvent	Yield (g)	Activity (kgPP/molM.hr)
Ziegler-Natta	Hexane	1.81	574
Ziegler-Natta	Toluene	2.11	670
Ziegler-Natta	Heptane	1.68	532
Ziegler-Natta	Xylene	1.94	616
Metallocene	Hexane	0.22	97
Metallocene	Toluene	3.08	1368
Metallocene	Heptane	1.36	603
Metallocene	Xylene	2.91	1293

a) Polymerization conditions: Ziegler-Natta: $[\text{Ti}] = 7 \times 10^{-5}$ M, $\text{Al}_{(\text{TEA})}/\text{Ti} = 167$, 40 °C, 40 Psi, 30 ml, 1.5 hr, metallocene: $[\text{Zr}] = 5 \times 10^{-5}$ M, $\text{Al}_{(\text{MAO})}/\text{Zr} = 2000$, 60 °C, 40 psi, 30 ml, 1.5 hr.

b) Ziegler-Natta: $\text{MgCl}_2/\text{TiCl}_4/\text{DEP-TEA}$, metallocene: $\text{SiO}_2/\text{MAO}/\text{rac-Et}(\text{Ind})_2\text{ZrCl}_2\text{-TMA}$.

Figure 4.1 shows the effect of solvent on the catalytic activity of two types of catalyst systems. In case of Ziegler-Natta catalyst, the activity of catalyst using aromatic solvents such as toluene and xylene is higher than those using aliphatic

solvents such as hexane and heptane. It is due to the better solubility of monomer in toluene and xylene than that in hexane and heptane[65]. When using metallocene catalyst, varying types of solvent affect to the activity of catalyst in the same way as Ziegler-Natta but much stronger. It is not due to only the effect of solubility of monomer but also the effect of solubility of the catalyst components. Metallocene catalysts exhibit high solubility in aromatic solvents, such as toluene, due to the cyclopentadienyl (Cp) rings of these solvents. Low polarity aliphatic hydrocarbons cannot be used as polymerization milieu because the solubility of catalyst components is low, MAO solubility is only 3-4 wt% in aliphatic solvents, and up to 30 wt% in aromatic solvents [65].

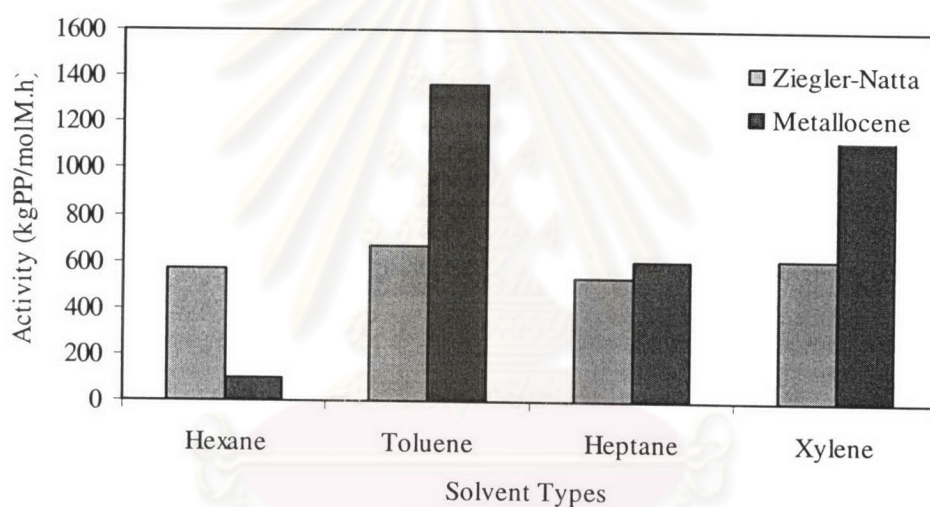


Figure 4.1 Catalytic activity using different types of solvent

4.1.2 The Effect of Solvents on the Isotacticity of Polymer

The isotacticity of polymer was influenced by the types of solvent used in polymerization. The result is illustrated in Table 4.2.

The effect of solvent on the isotacticity of polymer was displayed in Figure 4.2. From Figure 4.2 the isotacticity of polypropylene when using toluene and xylene as solvents was lower than that using hexane and heptane. It might be the effect of the solubility of diethylphthalate, internal donor, from catalyst into solvent. The internal donor that has the structure of aromatic should be better dissolved in aromatic solvent

than in aliphatic solvents. This assumption was proved by Infrared spectroscopy analysis of the liquid fraction obtained from the conditions using in polymerization in the absence of monomer. The IR spectrum was displayed in Figure 4.3.

Table 4.2 Isotactic index of polypropylene by Ziegler-Natta^{b)} catalyst using difference types of solvent^{a)}

Catalyst ^{b)}	Solvent	I.I. ^{c)} (%)	[mmmm] ^{d)} (%)	T _m ^{e)} °C
Ziegler-Natta	Hexane	70.5	68.5	152.43
Ziegler-Natta	Toluene	67.2	51.7	156.43
Ziegler-Natta	Heptane	70.4	65.9	156.43
Ziegler-Natta	Xylene	66.5	n.d.	n.d.
Metallocene	Hexane	n.d.	n.d.	n.d.
Metallocene	Toluene	n.d.	69.5	119.55
Metallocene	Heptane	n.d.	72.3	123.35
Metallocene	Xylene	n.d.	n.d.	n.d.

a) Polymerization conditions [Ti] = 7×10^{-5} M, Al_(TEA)/Ti = 167, 40 °C, 40 Psi, 30 ml, 1.5 hr,

b) Ziegler-Natta: MgCl₂/TiCl₄/DEP-TEA

c) Boiling heptane insoluble fraction.

d) Determined by ¹³C NMR spectroscopy of boiling heptane insoluble fraction.

e) Determined by DSC.

f) n.d. = not determined.

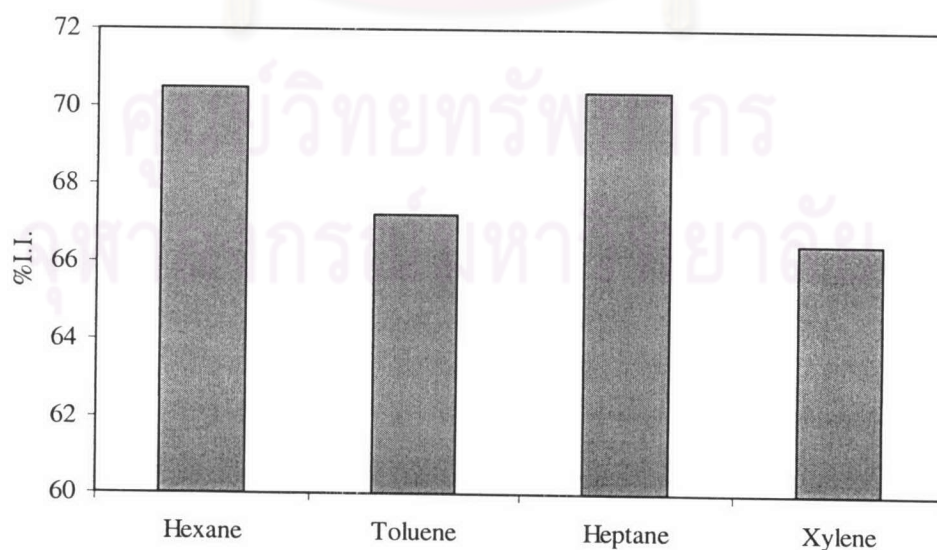
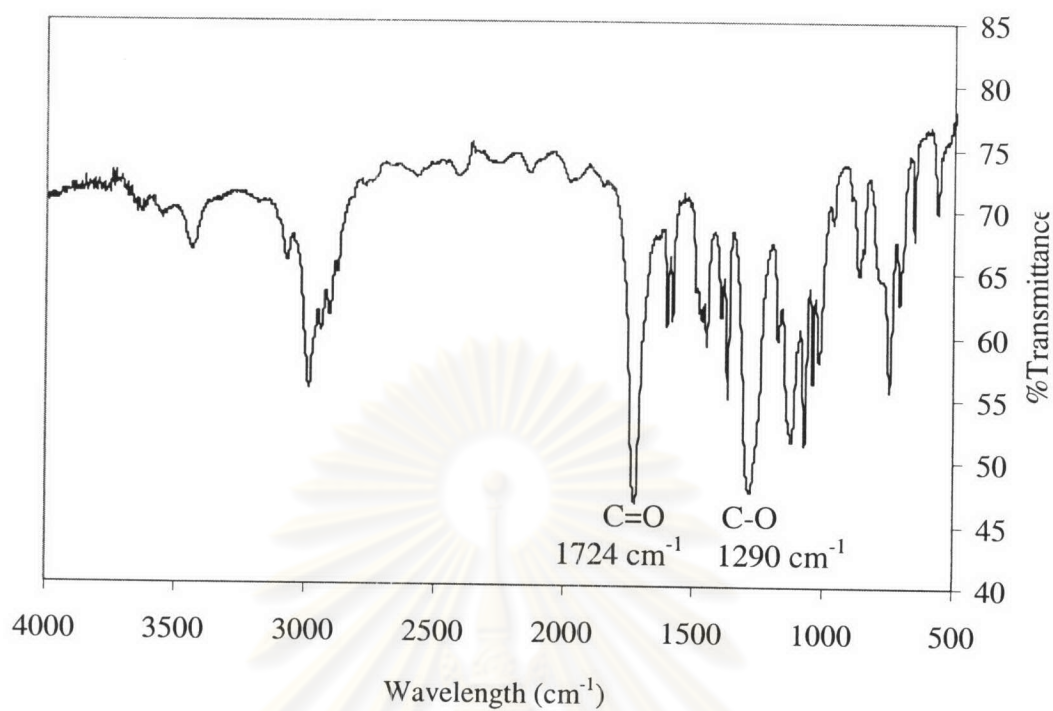
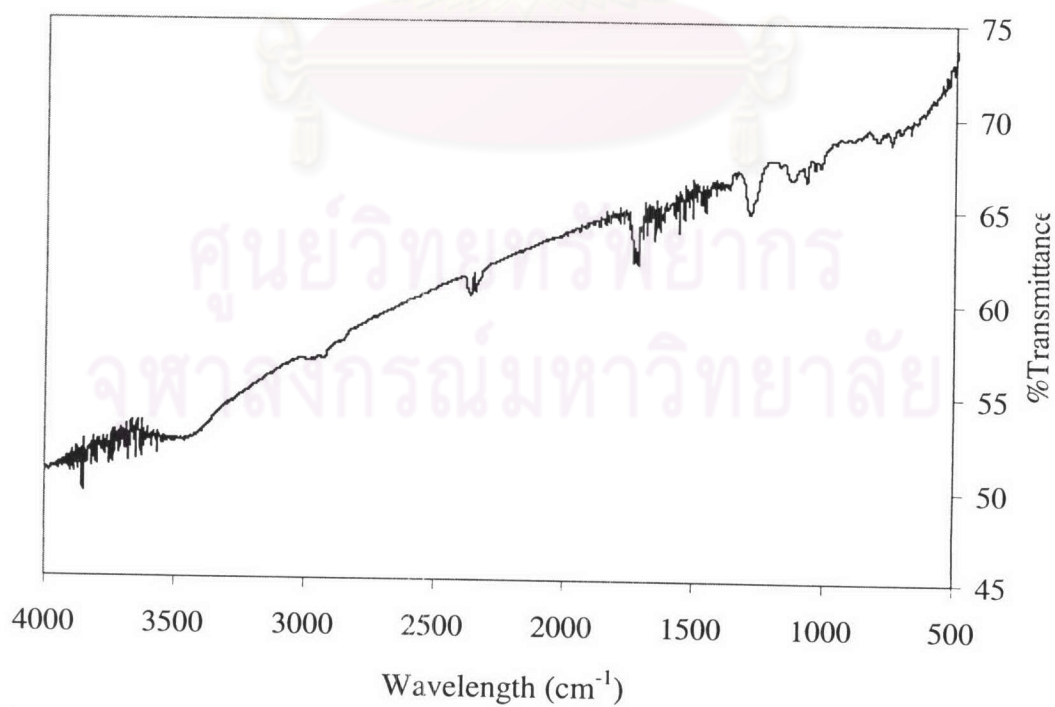


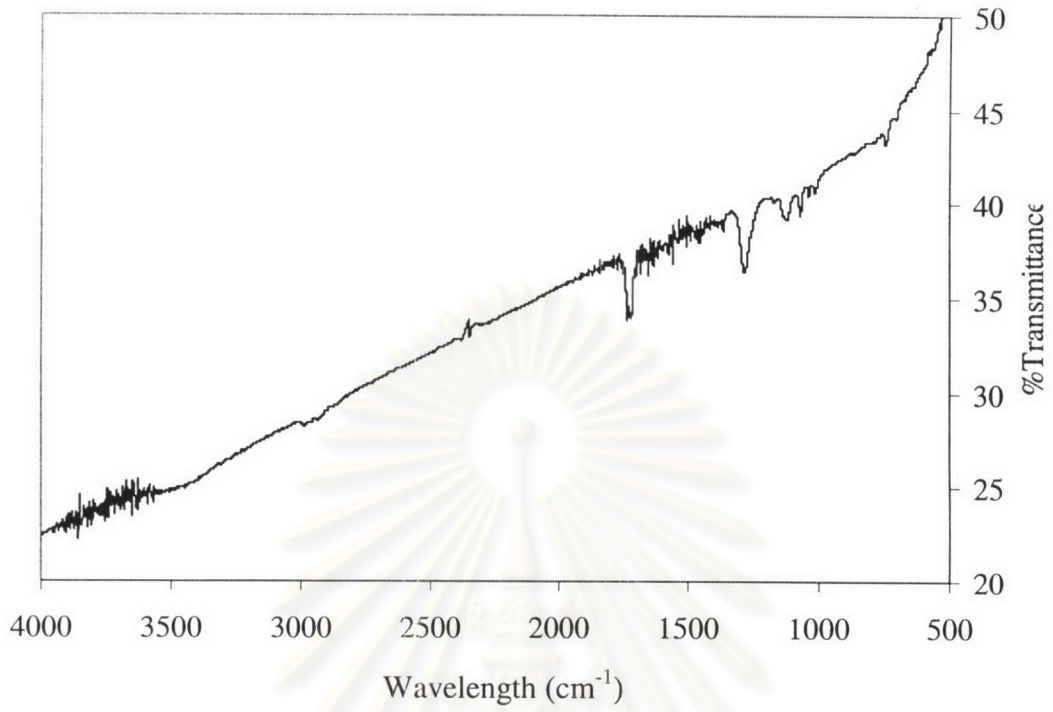
Figure 4.2 Isotactic index of polypropylene by Ziegler-Natta catalyst using different types of solvent



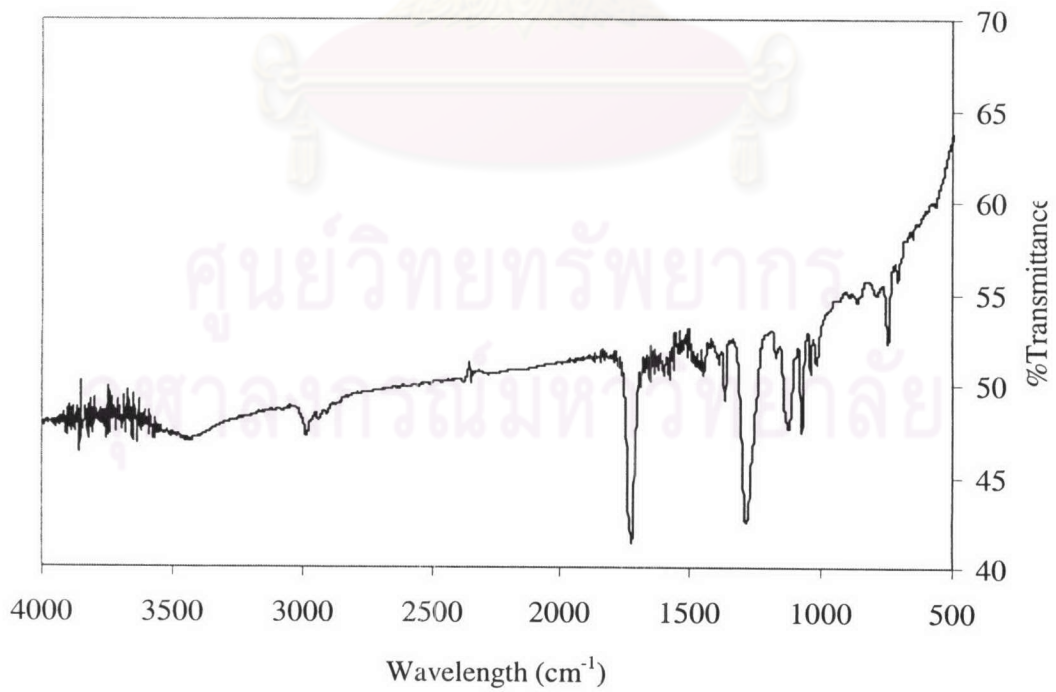
(A)



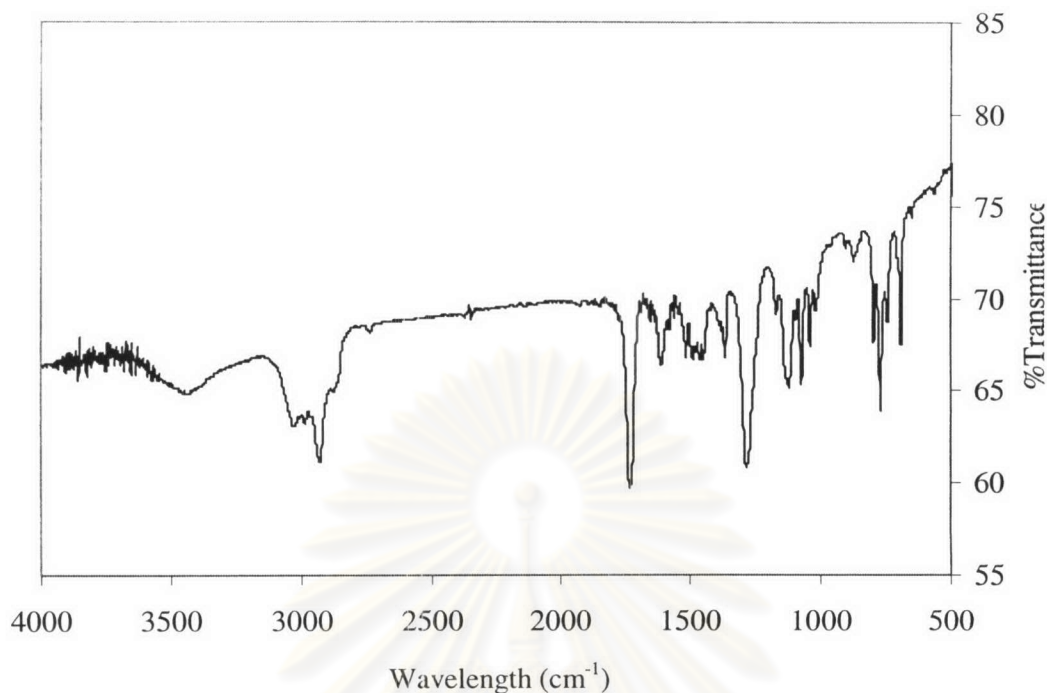
(B)



(C)



(D)



(E)

Figure 4.3 IR spectrum of DEP (A) and liquid fraction using various types of solvent: Hexane (B), Heptane (C), Toluene (D), Xylene (E)

From IR spectrum of diethylphthalate, the major bands were appeared at 1700 and 1300 cm^{-1} . It was cleared from the previous work [59] that it represented the coordination of C=O and C-O, respectively. The spectrum of liquid fraction using aromatic solvent; toluene and xylene, showed the major bands at the same positions observed from the spectrum of diethylphthalate (1700 and 1300 cm^{-1}) assigned to the C=O and C-O species in diethylphthalate, whereas the weaker peaks was observed when using aliphatic solvent, hexane and heptane, at the same positions.

Therefore, it can be summarized that diethylphthalate that use for improving the isotacticity of polymer could be dissolved in aromatic solvent better than in aliphatic solvent. It resulted in the lower isotacticity of polypropylene when using aromatic solvent than aliphatic solvent.

4.2 The Effect of Polymerization Temperatures

The effect of polymerization temperatures was investigated by propylene polymerization using two different catalyst system, supported Ziegler-Natta catalyst ($\text{MgCl}_2/\text{TiCl}_4/\text{DEP}$) and supported metallocene catalyst ($\text{SiO}_2/\text{MAO}/\text{TMA}/\text{rac-Et}(\text{Ind})_2\text{ZrCl}_2$). Series of propylene polymerization were performed at different temperatures in range of 40-70 °C.

4.2.1 The Effect of Polymerization Temperatures on the Catalytic Activity

Table 4.3 and Figure 4.4 illustrated the results for the effect of polymerization temperatures on the catalytic activity.

Table 4.3 Catalytic activity at different polymerization temperature ^{a)}

Catalyst ^{b)}	Temp (°C)	Yield (g)	Activity (kgPP/molM.hr)
Ziegler-Natta	40	3.03	963
Ziegler-Natta	50	3.10	985
Ziegler-Natta	60	3.25	1032
Ziegler-Natta	70	2.08	661
Metallocene	40	2.48	1103
Metallocene	50	5.61	2492
Metallocene	60	8.10	3600
Metallocene	70	4.79	2127

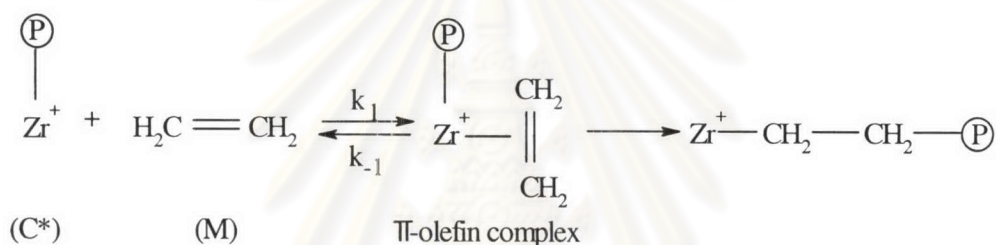
a) Polymerization conditions: Ziegler-Natta: $[\text{Ti}] = 7 \times 10^{-5}$ M, $\text{Al}_{(\text{TEA})}/\text{Ti} = 167$, 80 Psi, hexane 30 ml, 1.5 hr, metallocene: $[\text{Zr}] = 5 \times 10^{-5}$ M, $\text{Al}_{(\text{MAO})}/\text{Zr} = 2000$, 80 psi, toluene 30 ml, 1.5 hr.

b) Ziegler-Natta: $\text{MgCl}_2/\text{TiCl}_4/\text{DEP-TEA}$, metallocene: $\text{SiO}_2/\text{MAO}/\text{rac-Et}(\text{Ind})_2\text{ZrCl}_2\text{-TMA}$.

Figure 4.4 shows the variation in catalyst activities for different polymerization temperatures in the range from 40°C to 70°C, the activity of the catalyst increased in the range of 40-60 °C and decreased in the range of 60-70 °C.

In general, the rate of a chemically controlled reaction increases with temperatures as the rate constant is increased. In Ziegler-Natta catalysis, a widely accepted mechanism of propagation involves the initial formation of a π -complex of an olefin with the catalyst metal, followed by chain migratory insertion [60].

At low polymerization temperatures, the π -olefin complex is more stable; its lifetime is longer than the period of insertion. At high temperatures, the complex readily dissociates. The increase of the activity with the increased reaction temperatures may be attributed to the process of activated insertion and the activity is expected to decrease with further increased of reaction temperatures because of a shift of equilibrium to the dissociation of the π -complex [60].



From figure 4.4, the activity of metallocene catalyst is more sensitive to the increasing of polymerization temperature than that of Ziegler-Natta catalyst. It is due to more formation active site of metallocene catalyst than that of Ziegler-Natta catalyst, proposed by T Shiono.

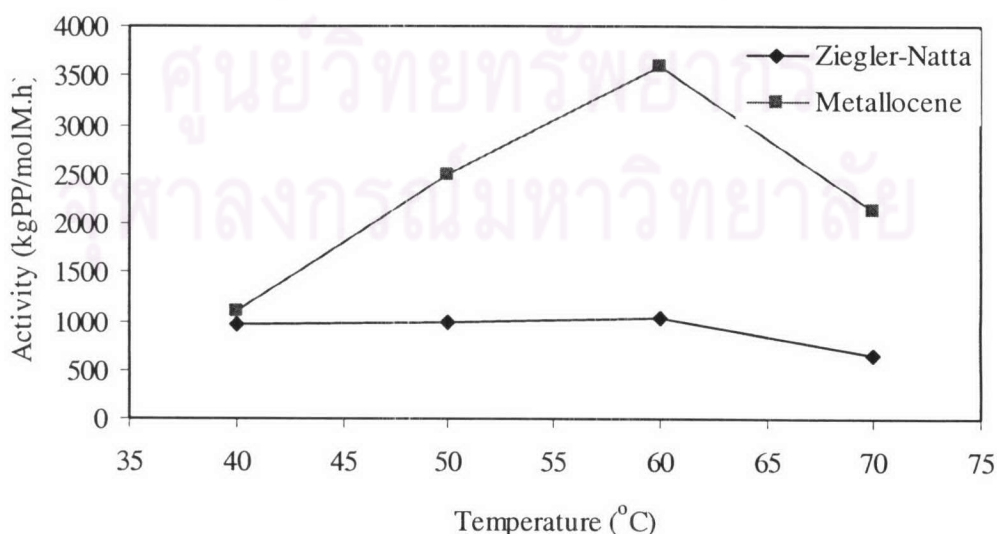


Figure 4.4 Catalytic activity at different polymerization temperature

4.2.2 The Effect of Polymerization Temperatures on the Isotacticity of Polymer

The effect of polymerization temperatures in propylene polymerization using two catalyst systems, Ziegler-Natta and metallocene, is shown in Table 4.4.

Table 4.4 Isotacticity of polypropylene at different polymerization temperatures ^{a)}

Catalyst ^{b)}	Temp (°C)	I.I. ^{c)} (%)	[mmmm] ^{d)} (%)	T _m ^{e)} (°C)
Ziegler-Natta	40	74.2	71.4	154.31
Ziegler-Natta	50	68.8	61.6	149.79
Ziegler-Natta	60	68.2	63.0	151.42
Ziegler-Natta	70	65.2	79.7	155.00
Metallocene	40	n.d.	74.8	129.01
Metallocene	50	n.d.	73.6	127.77
Metallocene	60	n.d.	71.9	123.85
Metallocene	70	n.d.	69.4	119.29

a) Polymerization conditions: Ziegler-Natta: [Ti] = 7×10^{-5} M, Al_(TEA)/Ti = 167, 80 Psi, hexane 30 ml, 1.5 hr, metallocene: [Zr] = 5×10^{-5} M, Al_(MAO)/Zr = 2000, 80 psi, toluene 30 ml, 1.5 hr.

b) Ziegler-Natta: MgCl₂/TiCl₄/DEP-TEA, metallocene: SiO₂/MAO/rac-Et(Ind)₂ZrCl₂-TMA.

c) Boiling heptane insoluble fraction.

d) Determined by ¹³C NMR spectroscopy of boiling heptane insoluble fraction.

e) Determined by DSC.

As illustrated in Figure 4.5, the increase in polymerization temperatures lead to a decrease in polypropylene isotacticity in temperatures range 40-70 °C using metallocene catalyst. It has been demonstrated [24] that for this type of C₂-symmetric metallocene the relatively poor stereoselectivity at elevated temperature is primarily due to a (much) larger ratio between the rates of monomer insertion and of chain epimerization rather than reduced enantioselectivity.

The effect of temperatures on a decrease in the polymer isotacticity was different when Ziegler-Natta catalyst is used. From Figure 4.5, the %mmmm was decreased in temperatures ranged 40-50 °C. For MgCl₂-supported TiCl₄ catalysts, many authors [56] supported the idea of the coexistence of two different isotactic site:

(1) low stereospecific sites featuring a Cl-vacancy that produce polymers of low isotacticity, and (2) high isospecific sites with a Lewis base attached that give a highly isotactic polymer. It is known that the internal Lewis base could be leached out into the polymerization medium by aluminum alkyl during polymerization [63]. The effectiveness of the leaching process increase with temperatures. Thus, it is understandable that the isotactic pentads decreased with increase temperatures. Temperature ranged 60-70 °C, %mmmm was increased with temperature. It is due to the temperature closed to the boiling point of hexane (69°C) resulting in evaporation of hexane. It makes catalyst concentration in the liquid phase higher responsible for the higher isotacticity of polypropylene as observed in section 4.4.2.

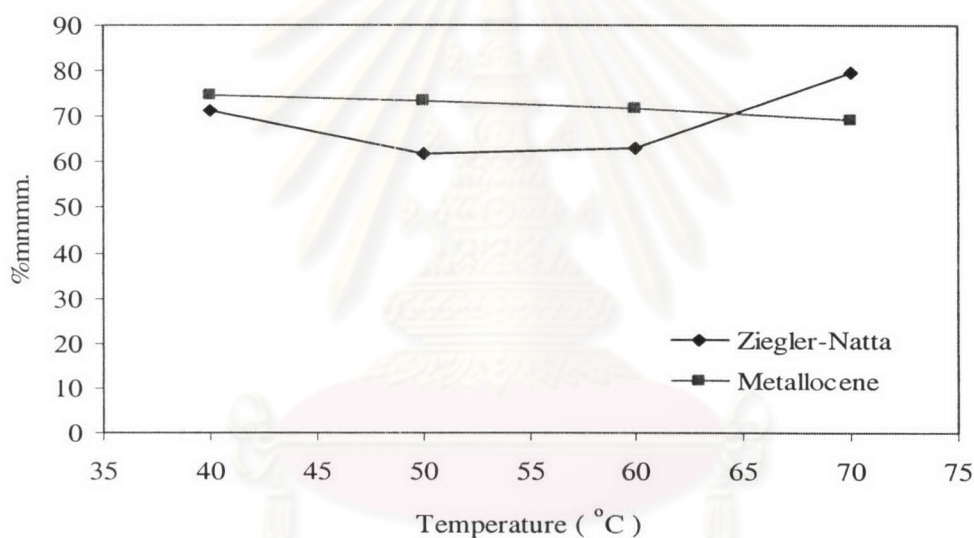


Figure 4.5 Isotacticity of polypropylene at different polymerization temperature

4.3 The Effect of Propylene Pressures.

Polymerization of propylene was conducted in 100 ml reactor for 90 minutes in range of pressure 40-100 psi using Ziegler-Natta and metallocene catalyst systems and the effect of propylene pressure was investigated.

4.3.1 The Effect of Propylene Pressures on Catalytic Activity

The effect of propylene pressure on catalytic activity was investigated in pressure range 40-100 psi. The polymerization results are shown in Table 4.5.

From Figure 4.6, the activity of both catalysts increased almost linearly with increasing propylene pressure. This is due to an increasing of propylene concentration in liquid phase resulting in increased activity in this pressure range. This behavior was described by Chien [63]. The propagation rate of propylene polymerization has been found to be $R_p = k_p[C^*][M]$. Thus, increasing in propylene pressures would result in an increase in the catalytic activity.

Table 4.5 Catalytic activity at different propylene pressures ^{a)}

Catalyst ^{b)}	Pressure (psi)	Yield (g)	Activity (kgPP/molM.hr)
Ziegler-Natta	40	1.81	574
Ziegler-Natta	60	2.34	742
Ziegler-Natta	80	3.03	963
Ziegler-Natta	100	3.37	1070
Metallocene	40	3.08	1368
Metallocene	60	5.57	2477
Metallocene	80	8.10	3600
Metallocene	100	10.53	4679

^{a)} Polymerization conditions: Ziegler-Natta: $[Ti] = 7 \times 10^{-5}$ M, $Al_{(TEA)}/Ti = 167$, 40 °C, hexane 30 ml, 1.5 hr, metallocene: $[Zr] = 5 \times 10^{-5}$ M, $Al_{(MAO)}/Zr = 2000$, 60 °C, toluene 30 ml, 1.5 hr.

^{b)} Ziegler-Natta: $MgCl_2/TiCl_4/DEP-TEA$, metallocene: $SiO_2/MAO/rac-Et(Ind)_2ZrCl_2-TMA$.

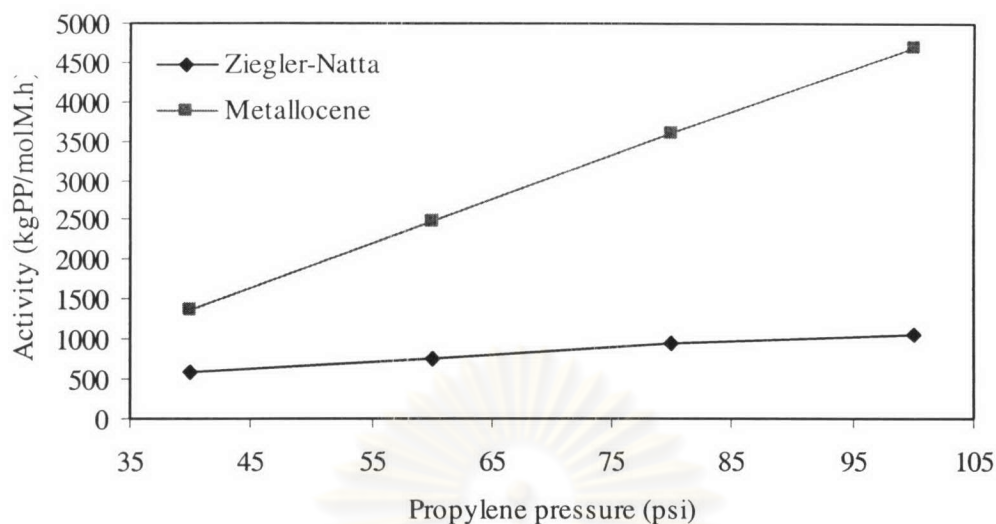


Figure 4.6 Catalytic activity at different propylene pressures.

4.3.2 The Effect of Propylene Pressures on the Isotacticity of Polymer

The effect of propylene pressure was investigated in pressure range 40-100 psi. The results were shown in Table 4.6.

Table 4.6 Isotacticity of polypropylene at different propylene pressure^{a)}

Catalyst ^{b)}	Pressure (psi)	I.I. ^{c)} (%)	[mmmm] ^{d)} (%)	T _m ^{e)} (°C)
Ziegler-Natta	40	70.5	68.3	152.43
Ziegler-Natta	60	72.3	68.6	150.35
Ziegler-Natta	80	74.2	71.4	154.31
Ziegler-Natta	100	76.8	71.0	153.43
Metallocene	40	n.d.	69.5	119.55
Metallocene	60	n.d.	71.5	121.45
Metallocene	80	n.d.	71.9	123.85
Metallocene	100	n.d.	77.1	125.30

a) Polymerization conditions: Ziegler-Natta: [Ti] = 7×10^{-5} M, Al_(TEA)/Ti = 167, 40 °C, hexane 30 ml, 1.5 hr, metallocene: [Zr] = 5×10^{-5} M, Al_(MAO)/Zr = 2000, 60 °C, toluene 30 ml, 1.5 hr.

b) Ziegler-Natta: MgCl₂/TiCl₄/DEP-TEA, metallocene: SiO₂/MAO/rac-Et(Ind)₂ZrCl₂-TMA.

c) Boiling heptane insoluble fraction.

d) Determined by ¹³C NMR spectroscopy of boiling heptane insoluble fraction.

e) Determined by DSC.

f) n.d. = not determined

In case of metallocene catalyst, %mmmm pentad was increased from 68.3% to 71.0% with the increase of propylene pressure in pressure range 40-100 psi. In recent papers, it has been shown that the stereoselectivity of propylene polymerization promoted by the class of C_2 -symmetric group 4 *ansa*-metallocene catalysts [24] is actually the result of an interplay of two competing reactions, namely isotactic monomer polyinsertion and a side process of epimerization of the polymer chain at its active end. A growing interference of the latter is responsible for the fade of stereoselectivity observed for most of these catalysts with decreasing monomer concentration. Whereas, %mmmm pentad of polypropylene preparing by Ziegler-Natta catalyst did not differ so much in the pressure range, suggesting that epimerization of growing polymer chain hardly occurs in Ziegler-Natta catalyst [64].

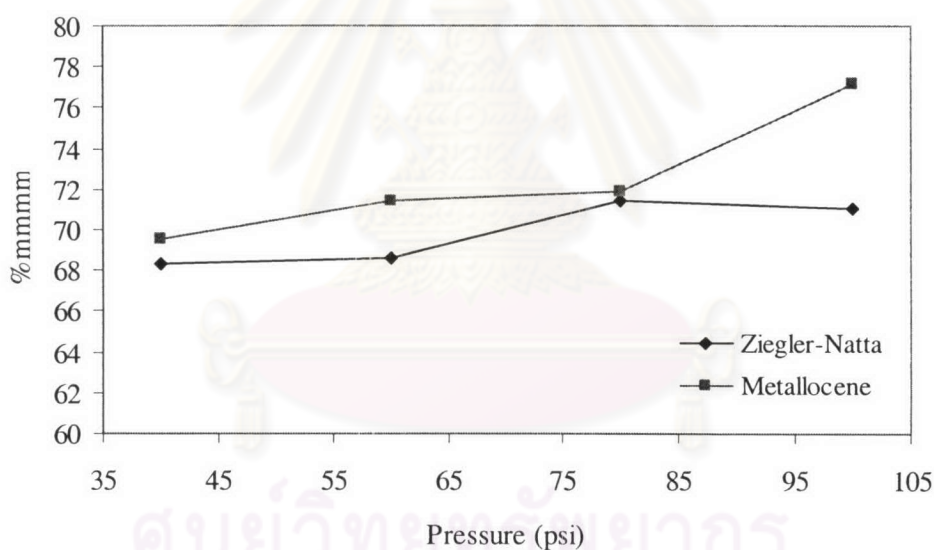


Figure 4.7 Isotacticity of polypropylene at different propylene pressures

4.4 The Effect of Catalyst Concentrations

The influence of catalyst concentration was investigated by propylene polymerization using two different catalyst system, supported Ziegler-Natta catalyst ($MgCl_2/TiCl_4/DEP$) and supported metallocene catalyst ($SiO_2/MAO/TMA/rac-Et(Ind)_2ZrCl_2$). Series of propylene polymerization was performed in 30 ml total solution for 90 minutes in the range of catalyst concentration 5×10^{-5} M to 8×10^{-5} M.

4.4.1 The Effect of Catalyst Concentration on the Catalytic Activity

The effect of catalyst concentration was observed via propylene polymerization using catalyst concentration in range of 5×10^{-5} M to 8×10^{-5} M. The results are illustrated in Table 4.7 and Figure 4.8.

Figure 4.8 presents the catalyst activity of Ziegler-Natta and metallocene catalysts that increases with increasing of catalyst concentrations in lower catalyst concentration regime and the activity was shown the highest at catalyst concentration of 6×10^{-5} M then decreases in the higher catalyst concentrations.

Table 4.7 Catalytic activity of different catalyst concentrations^{a)}

Catalyst ^{b)}	Catalyst Conc. ($\times 10^5$ M)	Yield (g)	Activity (kgPP/molM.hr)
Ziegler-Natta	5	0.97	429
Ziegler-Natta	6	1.58	584
Ziegler-Natta	7	1.81	574
Ziegler-Natta	8	1.93	537
Metallocene	5	3.08	1368
Metallocene	6	5.32	1969
Metallocene	7	6.09	1932
Metallocene	8	4.79	1329

a) Polymerization conditions: Ziegler-Natta: $Al_{(TEA)}/Ti = 167$, $40^\circ C$, 40 psi, hexane 30 ml, 1.5 hr, metallocene: $Al_{(MAO)}/Zr = 2000$, $60^\circ C$, 40 psi, toluene 30 ml, 1.5 hr.

b) Ziegler-Natta: $MgCl_2/TiCl_4/DEP-TEA$, metallocene: $SiO_2/MAO/rac-Et(Ind)_2ZrCl_2-TMA$.

The change of catalyst concentration should not result in the different nature of the active species formed. Only the number of active species should be increased according to the catalyst concentration. However, the dimerization of the active metal complexes forming an inactive species which illustrated in equation 4.1 and 4.2 favored at higher catalyst concentration is probably the explanation of this negative effect [61].

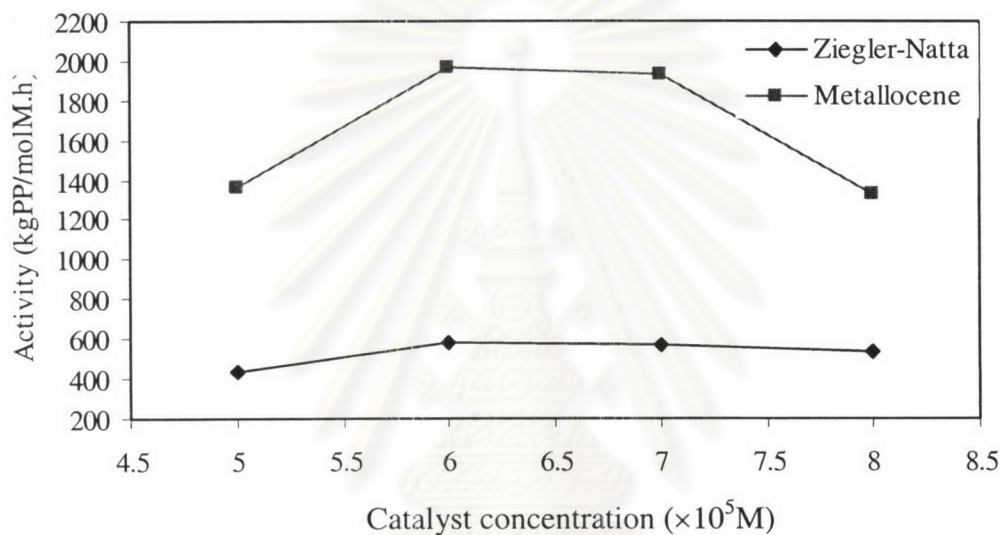
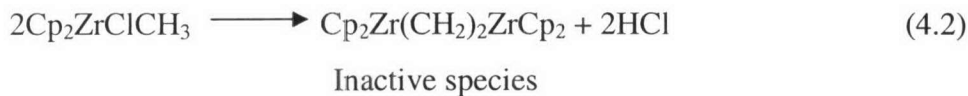
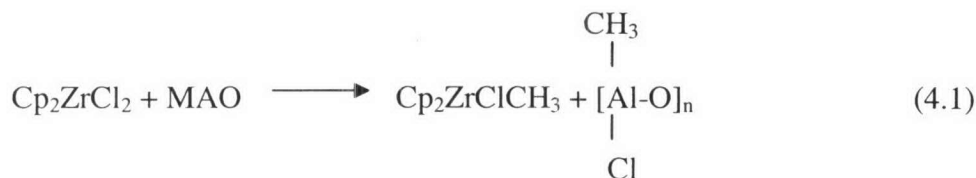


Figure 4.8 Catalytic activity of different catalyst concentrations

4.4.2 The Effect of Catalyst Concentration on the Isotacticity of Polymer

The effect of catalyst concentrations was observed via propylene polymerization using catalyst concentration in range of $5 \times 10^{-5} \text{ M}$ - $8 \times 10^{-5} \text{ M}$. The results was illustrated in Table 4.8.

In case of Ziegler-Natta catalyst, the %mmmm pentad was increased with increasing catalyst concentrations while, for metallocene catalyst, the %mmmm pentad of polypropylene did not change with the catalyst concentration range of $5 \times 10^{-5} \text{ M}$ - $8 \times 10^{-5} \text{ M}$ because the increase in Zr concentration did not effect to the nature of active sites that consistence with Busico [21], founding that the stereoselectivity of propylene polymerization promoted by C_2 -symmetric metallocene

catalyst is practically invariant when changing Zr concentration in the range 5×10^{-6} to 5×10^{-5} M.

Table 4.8 Isotacticity of polypropylene at different catalyst concentrations

Catalyst ^{b)}	Catalyst Conc. ($\times 10^5$ M)	I.I. ^{c)} (%)	[mmmm] ^{d)} (%)	Tm ^{e)} (°C)
Ziegler-Natta	5	69.7	46.9	152.05
Ziegler-Natta	6	70.2	46.9	152.53
Ziegler-Natta	7	70.5	68.3	152.43
Ziegler-Natta	8	69.5	71.0	154.31
Metallocene	5	n.d.	69.5	119.55
Metallocene	6	n.d.	67.8	118.45
Metallocene	7	n.d.	69.8	118.40
Metallocene	8	n.d.	72.4	120.77

a) Polymerization conditions: Ziegler-Natta: $\text{Al}_{(\text{TEA})}/\text{Ti} = 167$, 40 °C, 40 psi, hexane 30 ml, 1.5 hr, metallocene: $\text{Al}_{(\text{MAO})}/\text{Zr} = 2000$, 60 °C, 40 psi, toluene 30 ml, 1.5 hr.

b) Ziegler-Natta: $\text{MgCl}_2/\text{TiCl}_4/\text{DEP-TEA}$, metallocene: $\text{SiO}_2/\text{MAO}/\text{rac-Et}(\text{Ind})_2\text{ZrCl}_2\text{-TMA}$.

c) Boiling heptane insoluble fraction.

d) Determined by ^{13}C NMR spectroscopy of boiling heptane insoluble fraction.

e) Determined by DSC.

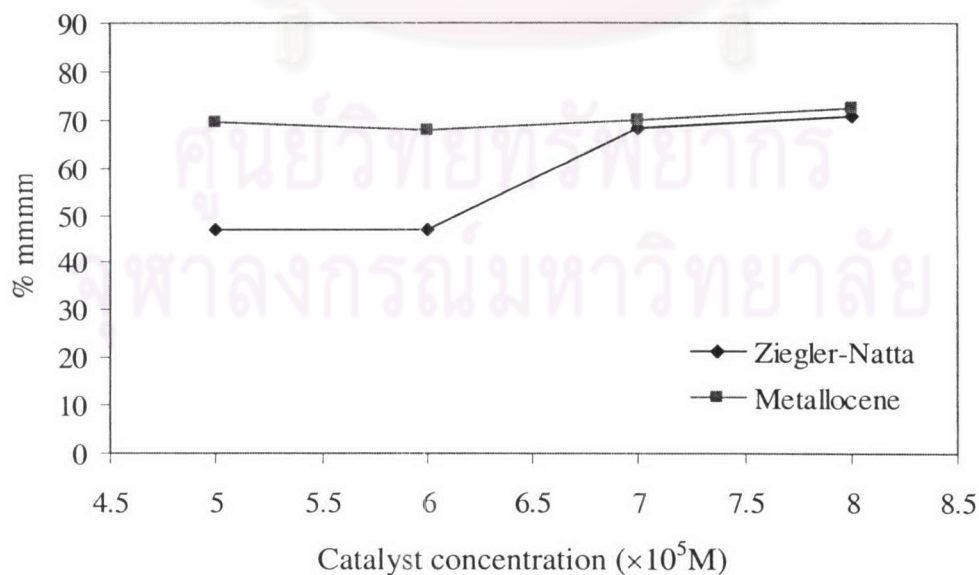


Figure 4.9 Isotacticity of polypropylene at different catalyst concentrations

Table 4.9 Propylene polymerizations using Ziegler-Natta catalyst

Catalyst Systems	Yield (kg PP/g Cat)	Insoluble Fraction (%)	%mmmm of insoluble fraction (%)
1. MgCl ₂ /TiCl ₄ /DEP-AlEt ₃ ^{a)}	0.9	%I.I. = 68.8	61.6
2. MgCl ₂ /TiCl ₄ /DIBP-AlEt ₃ ^{b)}	3	%X.I. = 73.3	81.7

a) Polymerization Conditions: [Ti] = 7×10^{-5} M, Al/Zr = 167, 40°C, 80 psi, hexane, 1.5 hr.

b) Polymerization Conditions: 10-20 g Cat, 800 mg AlEt₃, 1.3 kg Liquid PP, 40°C, 1 hr.

The Ziegler-Natta Catalyst prepared for this thesis (1) give lower activity than that of J. C. Chadwick (2) [76]. The obtained polypropylene has the lower % insoluble and %mmmm.

Table 4.10 Propylene polymerizations using metallocene catalyst

Catalyst Systems	Activity (kg PP/molZr h)	%mmmm (%)
1. SiO ₂ /MAO/rac-Et(Ind) ₂ ZrCl ₂ -TMA ^{a)}	2492	73.6
2. SiO ₂ /MAO/rac-Et(Ind) ₂ ZrCl ₂ -MAO ^{b)}	131	85

a) Polymerization Conditions: [Zr] = 5×10^{-5} M, Al/Zr = 2000, 50°C, 80 psi, toluene, 1.5 hr.

b) Polymerization Conditions: Al/Zr = 300, 50°C, toluene 30 ml, 1 hr.

The metallocene catalyst using in this thesis (1) give much higher activity than that of M. R. Ribeiro (2) [77]. The obtained polypropylene has the lower %mmmm.

4.5 Polymer Morphology

The morphologies of polypropylene obtained with Ziegler-Natta and metallocene catalysts using different types of solvent: heptane and toluene, were observed by scanning electron microscopy technique as shown in Figure 4.10.

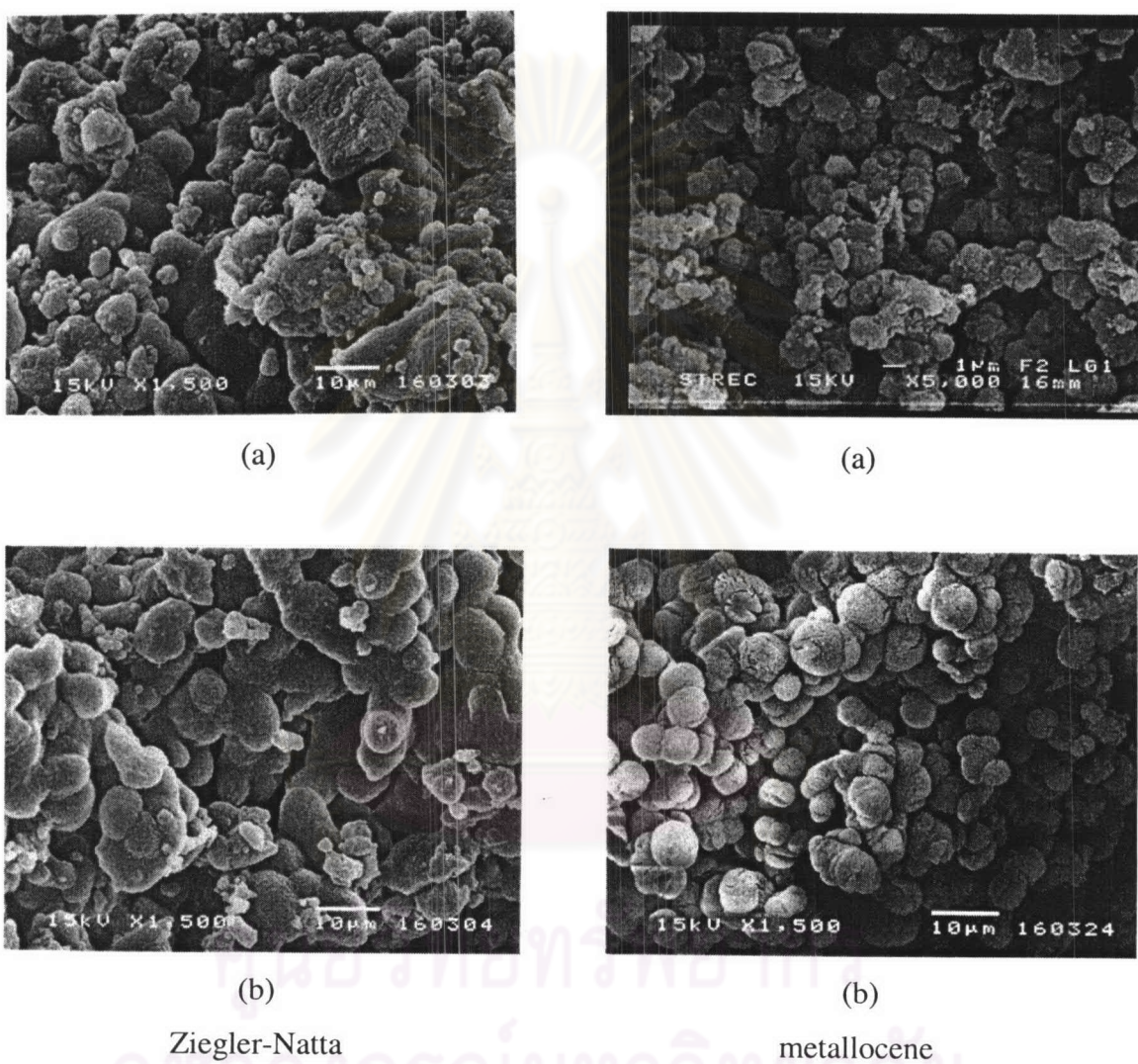


Figure 4.10 SEM pictures of polypropylene preparing by Ziegler-Natta and metallocene catalysts using different types of solvent: heptane (a) and toluene (b)

The morphologies of polypropylene obtained with Ziegler-Natta and metallocene catalysts at different catalyst concentrations: 5×10^{-5} and 7×10^{-5} M were observed by scanning electron microscopy technique as shown in Figure 4.11.

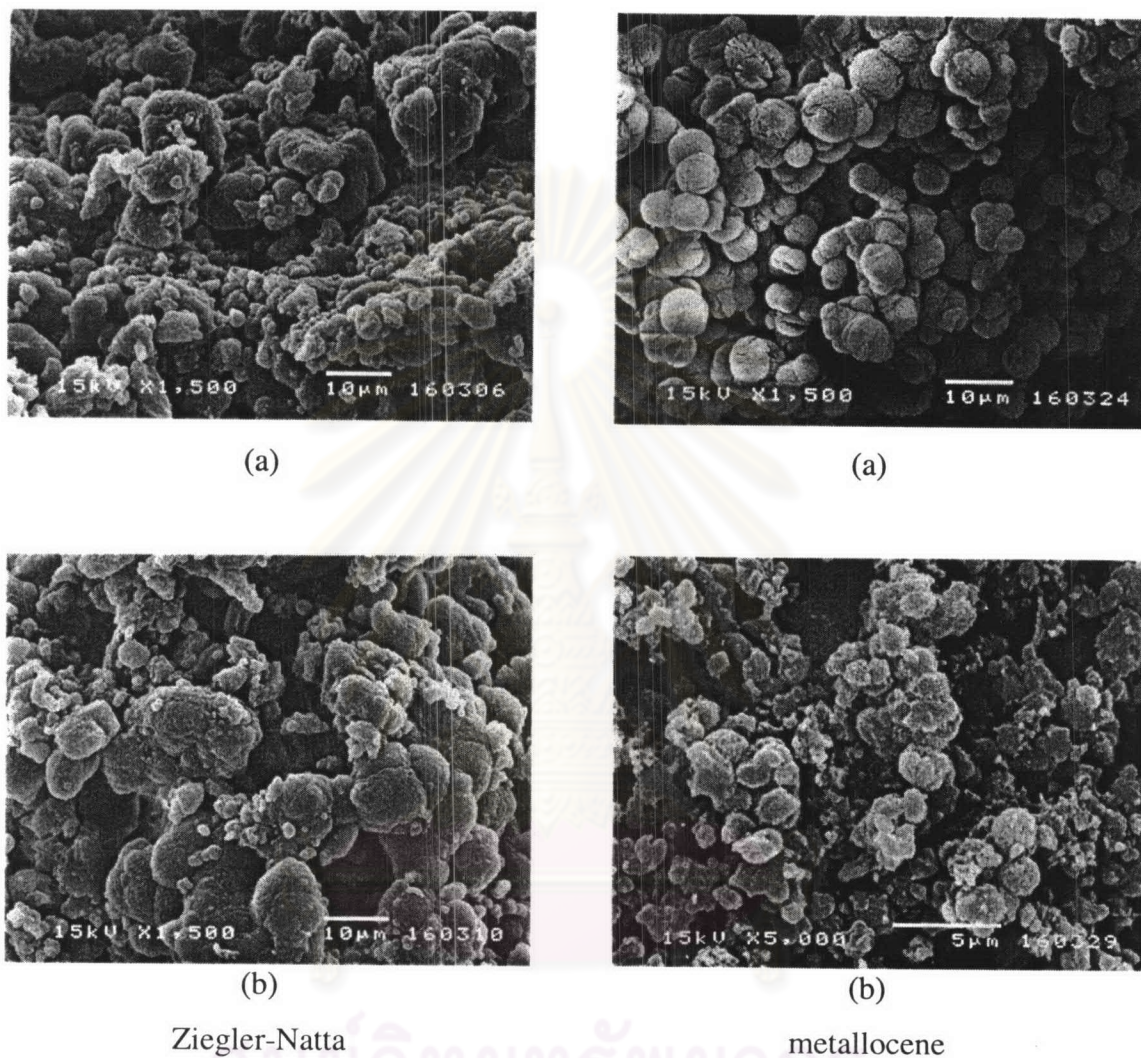


Figure 4.11 SEM pictures of polypropylene preparing by Ziegler-Natta and metallocene catalysts at different catalyst concentrations: 5×10^{-5} M (a) and 7×10^{-5} M (b)

Figure 4.12 shows the morphologies of polypropylene obtained with Ziegler-Natta and metallocene catalysts at different propylene pressure: 40 psi and 100 psi observed by scanning electron microscopy technique.

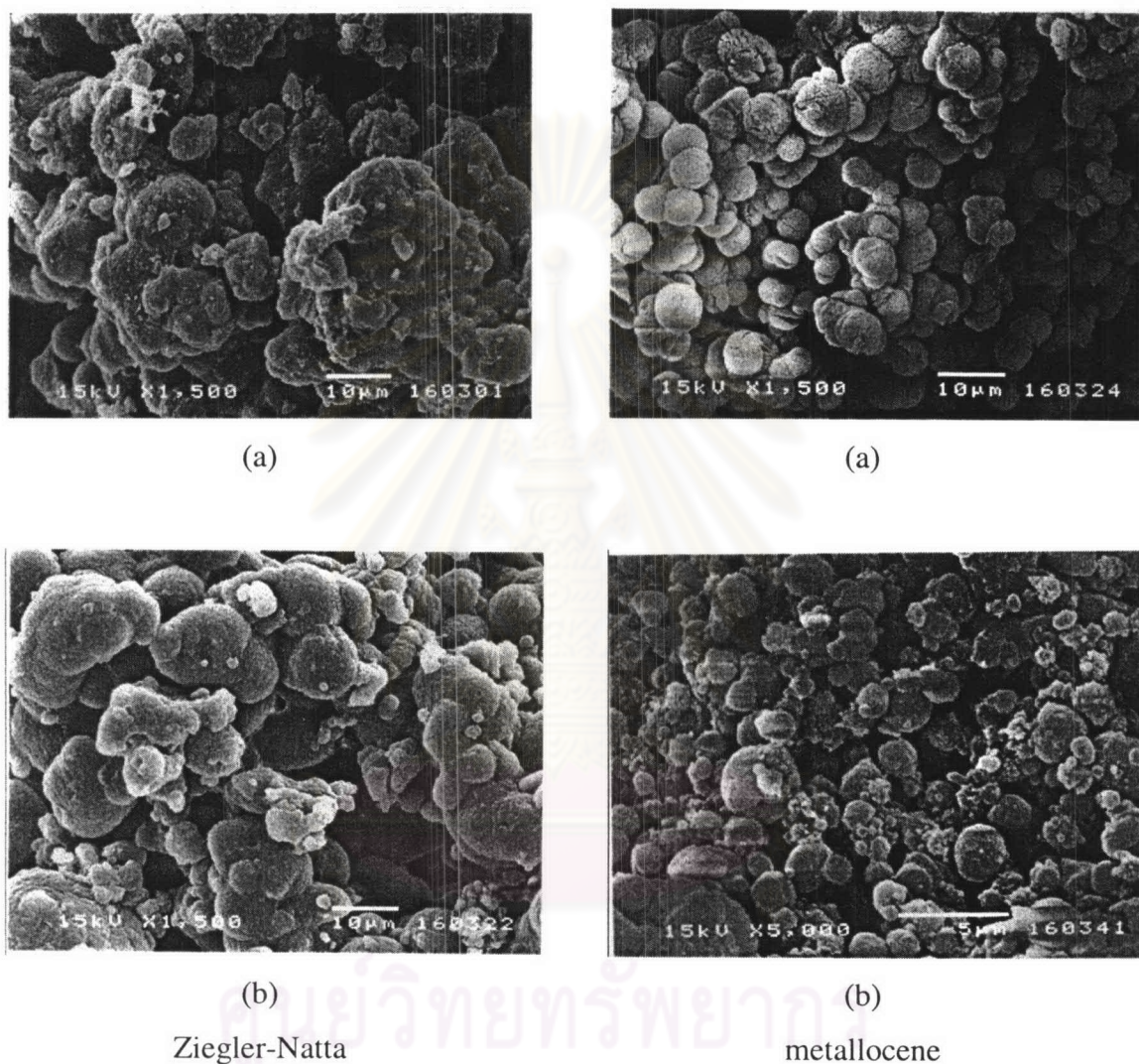


Figure 4.12 SEM pictures of polypropylene preparing by Ziegler-Natta and metallocene catalysts at different propylene pressure: 40 psi (a) and 100 psi (b)

Figure 4.13 shows the morphologies of polypropylene obtained with Ziegler-Natta and metallocene catalysts at different polymerization temperature: 40 °C and 80°C observed by scanning electron microscopy technique.

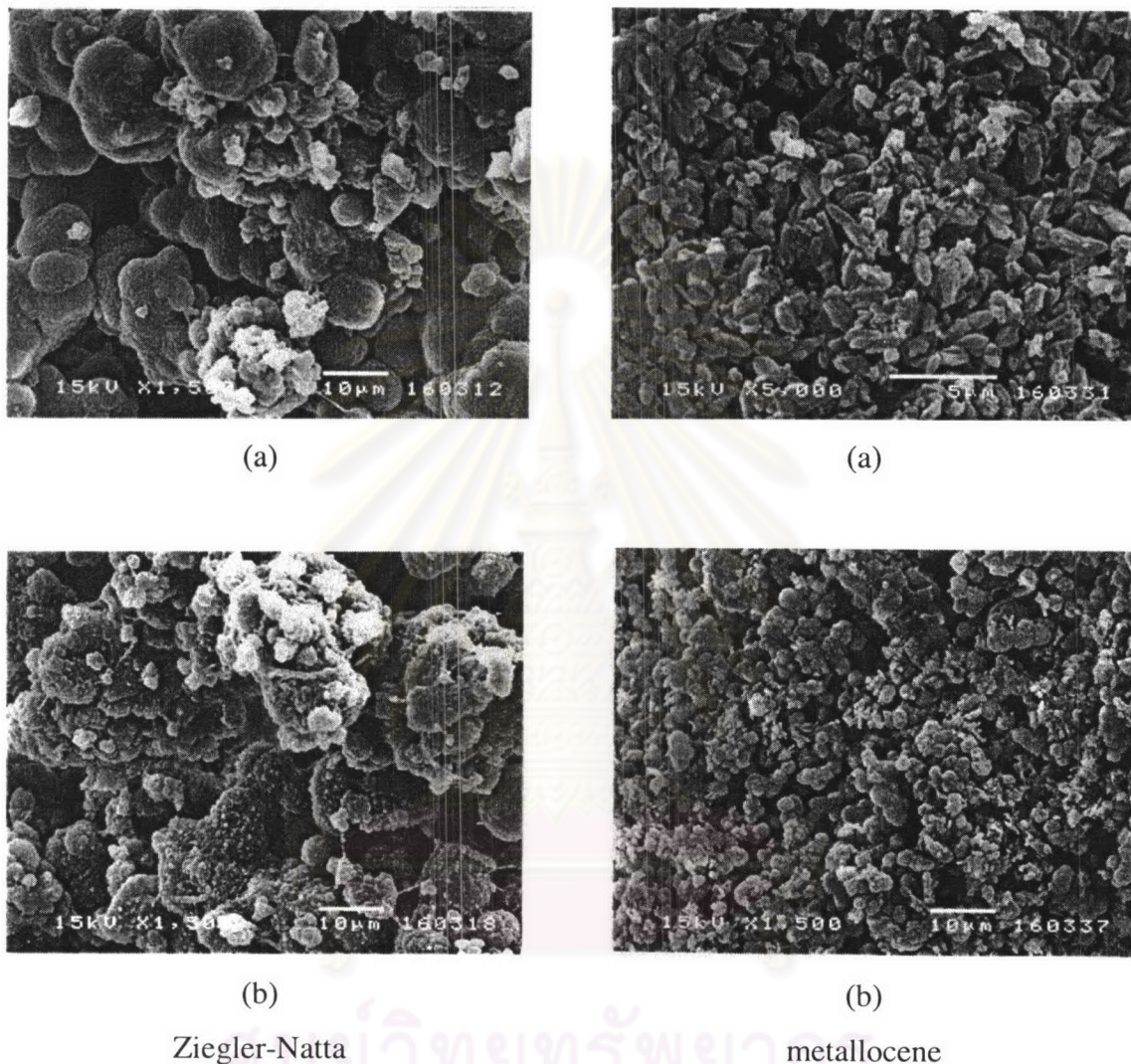


Figure 4.13 SEM pictures of polypropylene preparing by Ziegler-Natta and metallocene catalysts at different polymerization temperature: 40 °C (a) and 80°C (b)

It has been observed that the polymer particles produced by Ziegler-Natta and metallocene catalysts have a spherical morphology and does not change so much with polymerization conditions. In most conditions, polymer obtained from metallocene catalyst has a smaller size than that from Ziegler-Natta catalyst. It due the fragmentation of metallocene catalyst particles during polymerization.