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Propylene polymerization of *ansa*-complexes $(R^XPh)_2C(Cp)(Flu)MCl_2$ (M = Zr or Hf) with halogen substituents on phenyl groups

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Abstract

The catalytic properties of $(R^XPh)_2C(Cp)(Flu)MCl_2$ complexes (R^X = Cl, F or CF_3; M = Zr or Hf) in propylene polymerization were studied. The results showed dependences of stereospecificity and polymerization activity on the substituent on phenyl group. When R^X is CF_3, the activity is 1.38 times of that of the complex with no substituent at 1 atm propylene pressure and is extremely high at 3.0 MPa propylene pressure. The CF_3 group increased the regioirregular insertion during polymerization to produce partially crystalline s-PP. When R^X is halogen, the obtained polymer has higher melting point than that obtained from the complex with no substituent.
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1. Introduction

The bridged metallocene complexes are good catalyst precusors in stereospecific polymerization of α-olefins, and were well studied in the last decade. $Me_2C(Cp)(Flu)MCl_2$ (M = Zr, Hf) synthesized by Even et al. [1] were found highly active for polymerization of propylene to syndiotactic polypropylene. Then Razavi and Atwood [2] synthesized phenyl substituted bridged complexes $Ph_2C(Cp)(Flu)MCl_2$ (M = Zr or Hf) from which the syndiotactic polypropylene was obtained with high stereoregularity and high molecular weight. Since then many interests have been focused on metallocene dichloride structures with the bridged cylopentadienyl-fluorenyl ligand skeleton. The substituents on cylopentadienyl or fluorenyl strongly influence the catalytic activity and stereoselectivity [3–5]. There are only a few works concerning the influences of substituents on the phenyl groups of $Ph_2C(Cp)(Flu)MCl_2$ (M = Zr or Hf) on propylene polymerization. Kaminsky et al. [6,7] synthesized complexes $(MeOPh)_2C(Cp)$\{2,7-bis-(*t*-Bu)-fluorenyl\}MCl_2, $(CH_3Ph)_2C(Cp)$\{2,7-bis-(*t*-Bu)-fluorenyl\}MCl_2 (M = Zr or Hf) with electron donating groups (Me and MeO) and studied their catalytic behavior in propylene polymerization. Now we synthesized $(R^XPh)_2C(Cp)(Flu)MCl_2$ complexes (R^X = Cl, F or CF_3; M = Zr or Hf) and studied their catalytic behavior in propylene polymerization. It was found that the type and position of the substituent influenced not only the activity of the complex but also the tacticity of the polypropylene. Complex **1** with the CF_3 substituent at the *meta* positions on the phenyl groups showed the highest activity which was 1.36 times of that of $Ph_2C(Cp)(Flu)ZrCl_2$. The obtained polymer is syndiotactic polypropylene with lower tacticity than highly crystalline syndiotactic polypropylenes (thermoplastic material) from the conventional propylene polymerization catalyst $Ph_2C(Cp)(Flu)ZrCl_2$. The increases in atactic sequences can improve the machining, transparent and elastomeric properties of syndiotactic polypropylenes. It is known that some of the isotactic polypropylenes with atactic blocks have interesting elastomeric properties [8–11]. Elastomeric properties

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of polypropylene can be obtained by alternating ordered and disordered stereogenic blocks [12]. Syndiotactic polypropylene with lower tacticity may also have elastomeric properties [13,14]. Here, we introduced the halogen or CF_3 substituent to phenyl groups in the complex $(R^XPh)_2C(Cp)(Flu)ZrCl_2$ to improve the properties of product polypropylene.

2. Results and discussion

The bridged cylopentadienyl-fluorenyl ligand was prepared analogously to the literature [2,6]. After the condensation of cyclopentadiene with substituted (F, Cl, or CF_3) benzophenons in ethanol and sodium metal for reduction, the substituted diphenylfulvene was obtained. Then the fluorenyl lithium salt was allowed to react with the fulvene. By hydrolysis the ligand was precipitated from diethyl ether as white solids. The ligand was treated with two equivalents of *n*-butyl lithium in diethyl ether and reacted with the equivalent amount of zirconium tetrachloride or hafnium tetrachloride. The complexes **1**–**6** were obtained by recrystallisation from toluene (Fig. 1).

The complexes **1** and **2** have CF_3 substituents at the *meta* positions on the phenyl groups. Halogen is at the *para* position on the phenyl group in complexes **4**–**6**. Complex **3** was synthesized for comparision. They were used for polymerization of propylene in the presence of MAO. The results are given in Table 1.

From Table 1 we found they all showed high activities in propylene polymerization. Especially, the activity of complex **1** is as high as 3.9×10^7 g PP/mol M h at 3.0 MPa and 70 °C.

The substituent effects in metallocene catalysts have been incisively reviewed [15,16]. In the metallocenes $[2,7\text{-}X_2\text{-}fluCMe_2\text{-}Cp]ZrCl_2$, the electron-withdrawing substituent (halogen) is not so effective as the electron-donating substituent (*t*-Bu) in enhancing polymerization activity. Siedle et al. reported that the electron-withdrawing substituents should accelerate olefin polymerization because they rendered the metal center more electropositive. But a more electropositive metal center should have enhanced interactions with the counter ion of cocatalyst, leading to slower initialization and propagation [16].

The relative activity of complexes $(R^XPh)_2C(Cp)(Flu)ZrCl_2$ for propylene polymerization is in the order $R^X = CF_3 > H >$ halogen (F or Cl). At 1 atm propylene pressure, the activity of complex **1** is 26.0×10^5 g PP/mol M h which is 1.36 times of that of **3**. But the activities of complex with halogen substituents are much lower than **3**, which shows the electron-withdrawing effect is not in favor of polymerization. The electron-withdrawing substituent can make metal center more electropositive and form tighter ion pairing. This is not beneficial to polymerization in accordance with what Siedle et al. reported [16]. But the CF_3 substituent which is electron-withdrawing shows a quite different effect. So we think the position of substituent on the phenyl group plays a key role. The CF_3 substituent at the *meta* position is

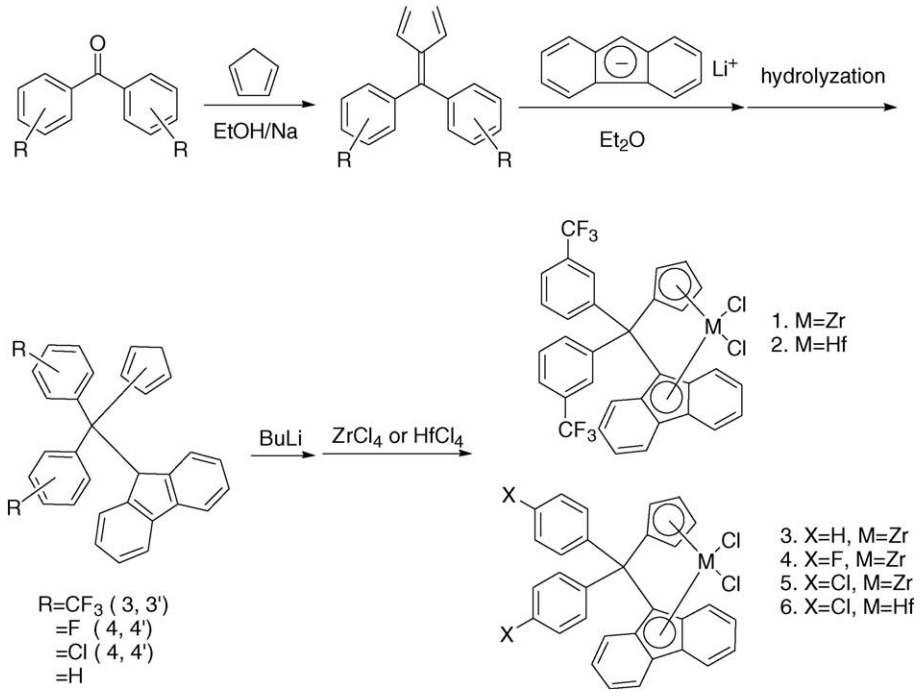

Fig. 1. Synthesis of complexes **1**–**6**

Table 1
The results of propylene polymerization with complexes **1**–**6**

Run no.	Complex	T_p (°C)	Activity[a] ($\times 10^{-5}$)	$M_\eta \times 10^{-5}$	T_m[b] (°C)	ΔH_f[c] (J/g)
1	**1**	30	26.0	2.46	120	24.6
2		50	19.6	1.49		
3		70	8.41	0.34		
4[d]		70	389		129	
5	**2**	30	2.18			
6		50	1.37			
7		70	0.88			
9	**3**	30	19.2	2.96	127	45.6
10		50	14.5			
11	**4**	30	7.11	2.46	137	38.5
12		50	6.90	0.86		
13		70	5.40	0.45		
14		0	2.26	2.48		
15	**5**	30	17.4		133	41.6
16		0	2.56			
17	**6**	30	1.27			

Conditions: $P_{propylene}$ = 1 atm, time = 0.5 h, [Al]/[M] = 1000, [M] = 0.5×10^{-4} mol/l, 50 ml toluene.
[a] g PP/mol M h.
[b] Melting peak temperature of the DSC curve.
[c] Endothermic enthalpies determined by DSC as a parameter of the crystallinity of polymer.
[d] $P_{propylene}$ = 3.0 MPa, time = 2 h.

closer to metal in space than halogen substituents at the *para* positions. The active center is shielded by six fluorine atoms and kept away from counter ion charge because the fluorine atoms and counter ion repel each other. Although the active center is more electropositive, the loose ion pairing should prevail and results in enhanced interactions between metal center and olefin. This is beneficial to the polymerization. There is also a possibility that the steric hindrance of CF_3 group leads to the concomitant changes of the structure of the complex, facilitating the coordination and insertion of the propylene in polymerization.

The activity of the catalyst depends on the polymerization temperature. The activities decrease with the rise in polymerization temperature from 30 to 70 °C. Complexes **1**–**6** show the highest activity at 30 °C.

The viscosity molecular weight (M_η) of polypropylene produced by complex **1** at 30 °C is almost the same as by complex **4**, and lower than that produced by complex **3**. It shows that the CF_3 substituents or the fluorine substituents accelerate the chain transfer rate during polymerization. The electron-withdrawing effect of the CF_3 or fluorine substituent makes the metal center more electropositive. It tends to catch β-H of growing chain or promote other chain transfer reactions. Ewen studied complexes $Me_2C(Cp)(Flu)ZrCl_2$ with halogen substituents on fluorene. They think the substituent withdraws the electrons away from fluorene, thus drastically decreasing its basicity (more electropositive). This resulted in low molecular weights of polymers [17].

The polymer from **3** is highly crystalline syndiotactic polypropylene with very high tacticity ([r] = 97.5%) [2] and relatively long average syndiotactic block length (Lsyn = 3 + 2 × rrrr/rrrm), for example, 50–200 monomer units [13]. Although there are halogen substituents at the *para* positions on the phenyl groups, the structure of the complex **4** remains Cs symmetrical. It also produces highly crystalline syndiotactic polypropylene with [rrrr] = 85.1% ([r] = 95.1%) and Lsyn = 50 as complex **3**. The polypropylene from **4** has even higher melting point than from **3** (137 °C > 127 °C) (Table 2).

The Cs symmetric structure of complex **1** was destroyed by the CF_3 group at the *meta* positions on the phenyl groups nonsymmetrically located. One of the CF_3 group bends toward cyclopentadienyl and the other bends toward fluorenyl according to ^{1}H NMR spectra data. This structure tends to be C_2 symmetrical. According to the ^{13}C NMR spectra of polymer from **1** the two consecutive isotactic placements (an [mm] stereodefect) remain constant, but isolated placement (an [m] stereodefect) increases when compared with polymer from **4**. The isolated [m] stereoerrors can arise from site epimerization (chain migration without insertion) [14]. We

Table 2
Pentad distributions for polypropylene from **1** and **4** at 30 °C

Run no.	Complex	rmmr (%)	mmrr (%)	rmrr (%)	rrrr (%)	rrrm (%)	mrrm (%)
1	**1**	2.0	3.9	8.9	74.1	6.4	4.7
11	**4**	1.6	3.5	3.0	85.1	3.4	3.4

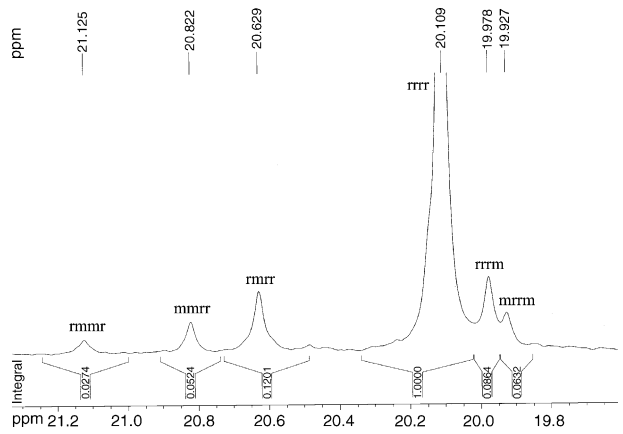

Fig. 2. ^{13}C NMR (methyl region) of syndiotactic polypropylene synthesized using 1/MAO at 30 °C.

think during polymerization when the polymer chain is at the side of CF_3 group bending toward cyclopentadienyl, the chain may tend to migrate to the side where CF3 group bends to fluorenyl. So the CF_3 groups increase the [rmrr] and [rrrm] pentads in polymer chain (Fig. 2).

The polypropylene from **1** has low tacticity ([r] = 91.6%) and short average syndiotactic block length (Lsyn = 26). As a result its melting point is low (120 °C) and ΔH_f is small (24.92 J/g) compared to the polymer obtained from the complex without substituent. Fig. 3 shows its DSC curve is broad. So it is not highly crystalline syndiotactic polypropylene, but partial crystalline polypropylene with low average syndiotactic block length. This may improve the elastomeric properties of the polymer. Average block length, as determined by a numerical integration of the pentads, has a great effect on the properties of the polymer. Relatively short average block lengths, i.e. 6–15 tend to occur in a flexible and rubbery polymer which exhibits good elastic properties as reported by Job [13]. In addition the partial crystalline polypropylene with low melting point and ΔH_f has good machining and transparent properties.

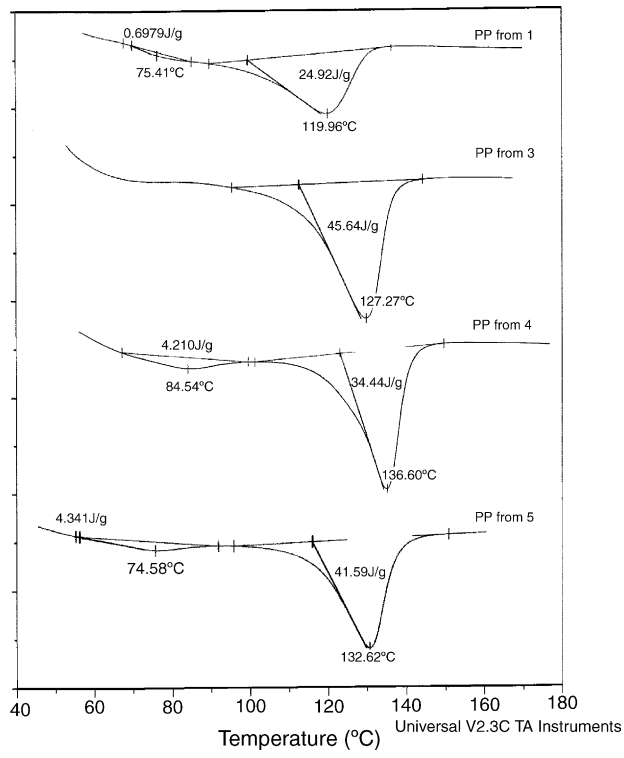

Fig. 3. DSC Curves of polypropylene obtained from complexes **1**, **3**, **4** and **5**.

3. Conclusions

In summary, the activity and stereoselectivity of complex $(R^XPh)_2C(Cp)(Flu)MCl_2$ (M = Zr, Hf) greatly depend on the type of R^X and the position of R^X on the phenyl group. The introduction of CF_3 at the *meta* position on the phenyl group can increase the activity in polymerization and decrease the tacticity and crystallinity of the polymer obtained. The complex with a halogen substituent at the *para* position showed low activity and produced polymers with high melting point.

4. Experimental

All the operations were carried out under a dry argon atmosphere using standard Schlenk techniques. Toluene, diethyl ether, and petroleum ether were refluxed over sodium/benzophenone ketyl, and distilled prior to use. The cocatalyst 1.53 M methylaluminoxane (MAO) in toluene was purchased from Witco.

Elemental analyses were carried out on an EA-1106 type analyzer. ^{1}H NMR were recorded on a Bruker AVANCE 500 MHz spectrometer with TMS as internal standard. MS spectra were recorded on a HP 5989A instrument. Differential scanning calorimetry was performed on a Universal V2.3C TA instrument at a heating rate of 10 °C/min. ^{13}C NMR spectra were recorded on a DR 500 Bruker spectrometer operating at 125.78 MHz in *o*-dichlorodeuterobenzene.

4.1. Preparation of 1,1′-(2,4-cyclopentadien-ylidenemethylene)bis-(3-trifluoromethyl-phenyl)

To a solution of sodium ethoxide prepared from adding 0.41 g (18.0 mmol) sodium to 20 ml ethanol were added 5.56 g (18.0 mmol) bis-(3-trifluoromethyl-phenyl) methanone and 1.48 ml (18.0 mmol) freshly prepared cyclopentadiene. Under good stirring for 10 min the yielded dark red solution was kept at 0 °C for 1.5 h. The product was then collected on a filter, washed with several portion of alcohol, and dried, to give a red crystal 4.78 g (yield, 73.4%). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 6.20 (m, 2H, Cp), 6.66 (m, 2H, Cp), 7.47 (d, J = 7.80 Hz, 2H, Ph), 7.54 (t, J = 7.80 Hz, 2H, Ph), 7.58 (s, 2H, Ph), 7.69 (d, J = 7.80 Hz, 2H, Ph).

1,1′-(2,4-cyclopentadien-ylidenemethylene)bis-(4-chloro-phenyl) and *1,1′-(2,4-cyclopentadien-ylidenemethylene)*

bis-(*4-fluoro-phenyl*) were obtained by using a similar procedure.

4.2. Preparation of complex **1**
[($C_{13}H_8$-μ-C(m-CF_3-Ph)$_2$-C_5H_4)$ZrCl_2$]

The solution of 6.0 mmol fluorenyl lithium salt in 20 ml ether was added dropwise to a solution of 1.59 g (6.0 mmol) *1,1′-(2,4-cyclopentadien-ylidenemethylene)bis-(3-trifluoromethyl-phenyl)* in 40 ml ether. After stirring for about 2 h, the solution was hydrolyzed by 20 ml water. The ligand as white solid 1.16 g was precipitated (yield, 45.3%).

To a solution of 1.16 g (2.2 mmol) ligand in 30 ml ether, 2.2 ml (4.4 mmol) *n*-butyllithium (2.0 M solution in *n*-hexane) was added dropwise at −78 °C. After stirring overnight, 0.51 g (2.2 mmol) $ZrCl_4$ was added and the solution was stirred for 8 h at room temperature, and then evaporated to dryness. The residue was recrystallized by toluene to give 0.90 g (yield, 49%) complex **1** as a red crystal.

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.24 (m, 2H, fluorenyl), 8.19 (m, 2H, Ph), 8.09 (m, 2H, Ph), 7.61–7.64 (m, 5H, Ph and fluorenyl), 7.53 (t, 1H, J = 8.14 Hz, Ph), 7.06 (t, 2H, J = 7.80 Hz, fluorenyl), 6.44 (m, 2H, fluorenyl), 6.28 (dd, 2H, J_1 = 11.01 Hz, J_2 = 2.03 Hz, Cp), 5.77 (dd, 2H, J_1 = 11.01 Hz, J_2 = 2.03 Hz, Cp). LRMS (70 eV): m/z = 690 (10, M^+), 530 (100, $\{C_{13}H_8$-μ-C(m-CF_3-Ph)$_2$-$C_5H_4\}^+$). Anal. calcd. for $C_{33}H_{20}Cl_2F_6Zr{\cdot}C_7H_8$: C, 61.21, H, 3.60; found: C, 61.62, H, 4.58.

4.3. Preparation of complex **2**
[($C_{13}H_8$-μ-C(m-CF_3-Ph)$_2$-C_5H_4)$HfCl_2$]· toluene

Complex **2** was synthesized by the procedure similar to that used for **1**.

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.15–8.23 (m, 4H, Ph and flurenyl), 8.09 (m, 2H, Ph), 7.57–7.66 (m, 5H, Ph and flurenyl), 7.54 (t, 1H, J = 8.20 Hz, Ph), 7.04 (t, 2H, J = 7.81 Hz, fluorenyl), 6.38 (m, 2H, fluorenyl), 6.23 (dd, 2H, J_1 = 10.05 Hz, J_2 = 2.02 Hz, Cp), 5.73 (dd, 2H, J_1 = 10.05 Hz, J_2 = 2.02 Hz, Cp). LRMS (70 eV): m/z = 778 (8, M^+), 530 (100, $\{C_{13}H_8$-μ-C(m-CF_3-Ph)$_2$-$C_5H_4\}^+$). Anal. calcd. for $C_{33}H_{20}Cl_2F_6Hf{\cdot}C_7H_8$: C, 55.09, H, 3.24; found: C, 54.81, H, 3.36.

4.4. Preparation of complex **3**
($C_{13}H_8$-μ-CPh_2-C_5H_4)$ZrCl_2$

Complex **3** was synthesized according to the literature [2].

4.5. Preparation of complex **4**
($C_{13}H_8$-μ-C(p-F-Ph)$_2$-C_5H_4)$ZrCl_2$

Complex **4** was synthesized by the procedure similar to that used for **1**.

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.22 (d, 2H, J = 8.37 Hz, fluorenyl), 7.88 (d, 2H, J = 7.44 Hz, Ph), 7.79 (d, 2H, J = 7.16 Hz, Ph), 7.60 (t, 2H, J = 8.37 Hz, fluorenyl), 7.48 (d, 2H, J = 7.44 Hz, Ph), 7.38 (d, 2H, J = 7.16 Hz, Ph), 6.92 (t, 2H, J = 8.60 Hz, fluorenyl), 6.42 (d, 2H, J = 8.60 Hz, fluorenyl), 6.40 (t, 2H, J = 2.61 Hz, Cp), 5.77 (t, 2H, J = 2.61 Hz, Cp). LRMS (70 eV): m/z = 511 (100, M^+ − Cl, –FC=CH), 430 (34, $\{C_{13}H_8$-μ-C(p-F-Ph)$_2$-$C_5H_4\}^+$), 426 (24, M^+ − fluorenyl). HRMS: calcd. for $C_{31}H_{20}Cl_2F_2Zr$: 589.9957; found: 589.9936.

4.6. Preparation of complex **5**
[($C_{13}H_8$-μ-C(p-Cl-Ph)$_2$-C_5H_4)$ZrCl_2$]·1.5 toluene

Complex **5** was synthesized by the procedure similar to that used for **1**.

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.22 (d, 2H, J = 8.46 Hz, fluorenyl), 7.85 (dd, 2H, J_1 = 8.43 Hz, J_2 = 2.41 Hz, Ph), 7.76 (dd, 2H, J_1 = 8.50 Hz, J_2 = 2.41 Hz, Ph), 7.60 (t, 2H, J = 8.46 Hz, fluorenyl), 7.45 (dd, 2H, J_1 = 8.43 Hz, J_2 = 2.34, Ph), 7.34 (dd, 2H, J_1 = 8.50 Hz, J_2 = 2.34, Ph), 7.07 (t, 2H, J = 8.10 Hz, fluorenyl), 6.44 (d, 2H, J = 8.10 Hz, fluorenyl), 6.40 (t, 2H, J = 2.68, Cp), 5.76 (t, 2H, J = 2.68 Hz, Cp). LRMS (70 eV): m/z = 622 (18, M^+), 511 (20, M^+ − p-Cl-Ph), 462 (100, $\{C_{13}H_8$-μ-C(p-Cl-Ph)$_2$-$C_5H_4\}^+$). HRMS: calcd. for $C_{31}H_{20}Cl_4Zr$: 621.9366; found: 621.9380.

4.7. Preparation of complex **6**
[($C_{13}H_8$-μ-C(p-Cl-Ph)$_2$-C_5H_4)$HfCl_2$]·1.5 toluene

Complex **6** was synthesized by the procedure similar to that used for **1**.

^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.19 (d, 2H, J = 8.35Hz, fluorenyl), 7.86 (dd, 2H, J_1 = 7.30 Hz, J_2 = 2.39 Hz, Ph), 7.76 (dd, 2H, J_1 = 7.35 Hz, J_2 = 2.39 Hz, Ph), 7.58 (t, 2H, J = 8.35 Hz, fluorenyl), 7.44 (dd, 2H, J_1 = 7.30 Hz, J_2 = 2.30 Hz, Ph), 7.34 (dd, 2H, J_1 = 7.35 Hz, J_2 = 2.30 Hz, Ph), 7.05 (t, 2H, J = 8.82 Hz, fluorenyl), 6.48 (d, 2H, J = 8.82 Hz, fluorenyl), 6.34 (t, 2H, J = 2.67 Hz, Cp), 5.71 (t, 2H, J = 2.67 Hz, Cp). LRMS (70 eV): m/z = 712 (100, M^+), 601 (82, M^+ − p-Cl-Ph), 462 (94, $\{C_{13}H_8$-μ-C(p-Cl-Ph)$_2$-$C_5H_4\}^+$). HRMS: calcd. for $C_{31}H_{20}Cl_4Hf$: 711.9785; found: 711.9725.

4.8. Polymerization procedure

A 100 ml flask was equipped with a propylene inlet, a magnetic stirrer, and a vacuum line. The flask was filled with 50 ml of freshly distilled toluene. MAO was added, and the flask was placed in a bath at the desired polymerization temperature for 10 min. The polymerization reaction was started by adding a solution of the catalyst precursor with a syringe. The polymerization was carried out for 0.5 h and then quenched with 3% HCl in ethanol (50 ml). The precipitated polymer was filtered and then dried overnight in a vacuum oven at 80 °C.

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