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Short communication

Chromium catalysts for ethylene trimerization/tetramerization functionalized with *ortho*-fluorinated arylphosphine ligand

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ABSTRACT

A series of novel asymmetric bidentate phosphines with *ortho*-fluorine substituents was developed and characterized. In combination with chromium, the compound gave high activity to 1-hexene and 1-octene (approaching 3200 kg/g Cr/h) and high combined selectivity (C_6-C_8 $>$ 95% in selected examples). With fluorine atoms at the *ortho*-position, the compound could be operable at a higher temperature without activity loss.

1. Introduction

Linear alpha-olefins (LAOs) are demanded as versatile raw materials for copolymers, plasticizers, detergents, and synthetic lubricants [1–6]. LAOs are generally produced with a broad range of olefins from C_4 to C_{20} featured by Schulz-Flory distributions manufactured by Shell, Ineos, SABIC, and Chevron-Phillips [7–9]. Among the LAOs, however, 1-hexene and 1-octene are the most significant alpha-olefins for the applications as the comonomers of the linear low-density polyethylene (LLDPE) which the demand is steadily increased. For this reason, producing 1-hexene and 1-octene selectively by trimerization and tetramerization of ethylene, plenty of researches have been carried out among the academic and industrial circles. Most widely known catalytic systems are the combination of bidentate phosphine ligands with chromium such as PNP [10–18], PN(C)$_n$NP [19], and PCCP [20,21]. BP, Sasol, and SKInnovation have developed independent technologies for the ethylene trimerization and tetramerization as the representatives in this industry. Although this selective oligomerization technology is currently considered as reaching the threshold of commercialization, some of the critical problems awaiting solution have remained. One of the process issues is that an inevitably generated high molecular polyethylene causes a severe line plugging into the process stream [22]. As an implementable solution, it is considered operating at a high temperature to dissolve generated polyethylene such as over 100 °C. To apply this skill, it must be secured to be superb catalytic stability at the high temperature; however, most of the conventional chromium based oligomerization catalysts could not afford to maintain their catalytic stabilities at higher reaction temperature because they often went through isomerization or degradation pathway [23,24]. Recently, some of *ortho*-functionalized aryl phosphine ligands were introduced to enhance catalytic performances on the oligomerization reaction. Sasol patented withdrawing groups such as -F, -OCF_3, -SCF_3 on the *ortho*-aryl phosphine of the PNP ligand systems (*e.g.*, (Ph$^{o\text{-}F}$)(Ph)PN(iPr)PPh$_2$ with a productivity of 1900 kg/g Cr/h and a selectivity of towards 56% of 1-hexene and 30% of 1-octene at 45 bar of ethylene, 100 °C in 27 mins of retention time) [25,26]. In our recent work, it was reported that the *ortho*-fluorinated aryl phosphine substituent increases catalytic activity especially at the high temperature using a POSS (polyhedral oligomeric silsesquioxane) introduced PNP ligand system (*e.g.*, (Ph$^{o\text{-}F}$)(Ph)PN(propylPOSS)PPh$_2$ with an activity of 1619 kg/g Cr/h and a selectivity of towards 37% of 1-hexene and 43% of 1-octene at 30 bar of ethylene, 100 °C in 15 mins of retention time) [22]. However, the reaction time was not enough to properly evaluate the activity of the catalyst, and also the role of fluorine atom could not be accurately investigated. Besides, it stimulates one's curiosity what would happen if the concept of *ortho*-fluorinated aryl phosphine ligand applied to PCCP ligand structures which are known for the more active structure than the PNP ligand system [20,21]. Consequently, further investigation was carried out on the PCCP ligand system manipulating a bulkiness of ligand backbone and controlling the number of the *ortho*-fluorinated aryl moiety.

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2. Experimental section

2.1. General conditions

All reactions were performed under an inert atmosphere using standard Schlenk techniques. All solvents and gases were dried and degassed using standard procedures. Chemicals were purchased from Sigma Aldrich or Alfa Aesar and used without further purification unless otherwise stated. mMAO-3A was obtained from Akzo Nobel Corporation as a 7% *w*/w solution in heptane. ^{1}H, ^{13}C, ^{19}F, and ^{31}P nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AVANCE III HD 500 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal tetramethylsilane signal at 0.0 ppm and the internal chloroform signal at 77.0 ppm as a standard. Elemental analyses were performed with a CE Instruments/ThermoQuest Italia Flash EA 1112 Series. Bruker Daltonics (Billerica, MA, USA) APPI 7 T FT-ICR MS was used for (+) mode atmospheric pressure photoionization analysis. Quantitative chromatographic analysis of the oligomerization products was performed using an Agilent 7890A GC-FID with an HP-PONA column (50 m × 0.20 mm). The reaction solvent, methylcyclohexane, was used as an internal standard. GPC analyses were performed on a 1260 Infinity II GPC/SEC system equipped with a dual flow refractive index (DRI) detector and a UV detector. The samples were analyzed in 1,2,4-trichlorobenzene at 145 °C using a flow rate of 1.0 mL/min. All polymers were injected at a concentration of 1 mg/mL in 1,2,4-trichlorobenzene, after filtration through a 2.5 μm syringe filter. The average molar masses (number average molar mass Mn and weight average molar mass Mw) and the molecular weight distribution (Mw/Mn) were derived from the RI signal by a calibration curve based on polystyrene standards. DSC was performed using a TA DSC model Q200. The samples were analyzed under a nitrogen atmosphere according to the following cycles: in the first cycle, the sample was heated from 30 to 200 °C, at a heating rate of 10 °C/min, leaving the material at 200 °C for 1 min; the second cycle was done using a cooling rate of 10 °C/min, until 20 °C; in the third cycle, the sample was heated from 30 to 200 °C, at a heating rate of 10 °C/min.

2.2. General procedure for the synthesis of ligands

A series of novel ligands were obtained by suitable modification of literature methods (Scheme 1) [21,22]. n-Butyl lithium solution (26.6 mL, 42.5 mmol, 1.6 M in hexane) was slowly added to a solution of alkyne (50.0 mmol) in diethyl ether (42 mL) stirred at −78 °C under nitrogen condition for 1 h. Chlorodiarylphosphine (38.3 mmol) was slowly added and then stirred further 2 h. The mixture was naturally raised to the room temperature and filtered through a silica pad and dried *in vacuo*. Further purification was carried out by distillation or column chromatography. A portion of synthesized alkynyl diaryl phosphine compound A (0.5 mmol), copper(I) iodide (5 mol%) and cesium carbonate (10 mol%) were dissolved in *N,N*-dimethylformamide (1 mL) followed by dropwise of diarylphosphine (0.6 mmol, 1.2 eq), then the mixture was stirred for 3 h after raised temperature to 90 °C. After the reaction, volatile was removed by vacuum drying, and the mixture was cooled to room temperature and diluted to n-hexane: ethyl acetate (9:1 *v*/v) solution followed by silica column chromatography. A pure oil product $Ar_2PC(R) = CPAr_2$ could be obtained and analyzed by NMR and HR-MS. Yields and characterization data are summarized in Supplementary Information.

2.3. Preparation of chromium complexes

In a glovebox, each ligand (0.2 mmol) and $CrCl_3(THF)_3$ (0.2 mmol) are charged in a flask followed by 2.0 mL of dichloromethane, and then the solution was stirred for 1 h. The color of the reaction mixture changed from purple to blue. After this, the solvent was removed under reduced pressure, and the resultant solid was washed twice with 5 mL of n-hexane. The product was dried under vacuum to yield chromium complex. As followed in the previous literature, ligated chromium complexes tend to form bimetallic structures maintaining the oxidation state +3 [20]. The chromium complexes were characterized and summarized in Supplementary Information.

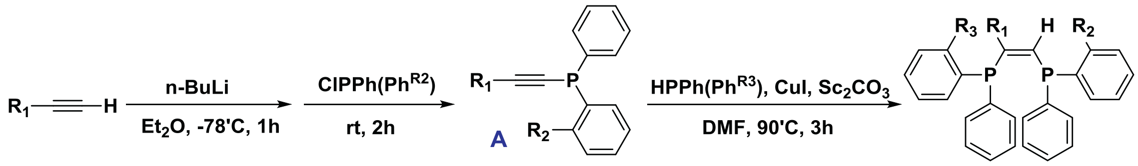
2.4. Ethylene oligomerization

All runs for ethylene oligomerization were carried out in a 3 L of Büchi stainless steel autoclave charged with methylcyclohexane (1 L) and mMAO-3A (500 equivalent of Al/Cr molar ratio), then heated to just below the desired reaction temperature. A synthesized chromium complex (suspension of solution in 5 mL of methylcyclohexane) was added to the autoclave, which was then pressurized with ethylene and stirred at 600 rpm. Ethylene was fed on demand to keep the reactor pressure constant, and the uptake was monitored using a mass flow meter (MFC). After an hour, the autoclave was cooled to 0 °C and depressurized slowly to atmospheric pressure. The product was quenched by adding 2-ethyl hexanol (2.0 mL). The crude products were filtered and analyzed using GC-FID (Typical GC-FID data in Fig. S1). The polymeric products were recovered by filtration and dried overnight in an oven at 100 °C.

3. Results and discussion

3.1. Synthesis of catalysts and their catalytic activity screening result

In our previous work, it was found that catalytic activity was maintained when the fluorine atom was introduced at the *ortho*-position on aryl phosphine ligand even at the high temperature over 140 °C without any thermally degradable signals such as excess polymer formation or deactivation [22]. From the result, some changes of the design were carried out on the ligand structure manipulating a bulkiness on the ligand backbone and introducing *ortho*-fluorine atoms according to a number or a position. Total 14 ligand structures and precatalysts derived from $CrCl_3(THF)_3$ were synthesized. After formation of the metal complex, *ortho*-fluorine atoms would be expected to be able to directly or indirectly affect the metal center during oligomerization



L1. R_1=nBu, R_2=R_3=H
L2. R_1=nBu, R_2=H, R_3=F
L3. R_1=nBu, R_2=F, R_3=H
L4. R_1=nBu, R_2=R_3=F
L5. R_1=iPr, R_2=R_3=H
L6. R_1=iPr, R_2=H, R_3=F
L7. R_1=iPr, R_2=F, R_3=H
L8. R_1=iPr, R_2=R_3=F
L9. R_1=Ph, R_2=R_3=H
L10. R_1=Ph, R_2=H, R_3=F
L11. R_1=Ph, R_2=F, R_3=H
L12. R_1=Ph, R_2=R_3=F
L13. R_1=tBu, R_2=R_3=H
L14. R_1=tBu, R_2=F, R_3=H

Scheme 1. Synthesis of asymmetric $Ar_2PC(R) = CPAr_2$ ligands.

Table 1
Ethylene oligomerization result.[a]

Ligand		Productivity (kg/g Cr/h)	C_6 (%)	1-C_6/C_6 (%)	C_8 (%)	1-C_8/C_8 (%)	C_{10-14} (%)	Polymer (%)	1-C_8/1-C_6
L1	60 °C	693	26.9	65.2	65.5	96.3	4.2	3.4	3.6
	100 °C	523	54.9	91.1	37.1	95.1	4.4	3.7	0.7
L2	60 °C	2204	40.2	84.4	55.6	97.4	3.7	0.5	1.6
	100 °C	2140	70.2	96.8	24.9	83.7	4.0	0.9	0.3
L3	60 °C	1172	41.0	83.0	52.7	98.8	4.3	2.0	1.5
	100 °C	1142	71.5	95.6	21.9	97.2	4.5	2.1	0.3
L4	60 °C	1256	44.7	84.3	43.0	97.5	3.1	9.2	1.1
	100 °C	1241	55.8	98.4	8.9	94.5	3.3	32.0	0.2
L5	60 °C	1643	33.4	82.8	62.5	97.8	3.6	0.5	2.2
	100 °C	1156	57.7	88.8	37.9	96.7	3.9	0.5	0.7
L6	60 °C	3328	45.0	85.8	50.1	98.9	4.5	0.4	1.3
	100 °C	3246	75.4	97.6	19.4	97.3	4.7	0.5	0.3
L7	60 °C	1293	45.3	84.3	51.1	96.2	3.3	0.3	1.3
	100 °C	1278	74.4	95.8	21.7	94.5	3.5	0.4	0.3
L8	60 °C	2921	52.1	88.9	44.2	99.7	3.2	0.5	1.0
	100 °C	2914	84.1	98.6	11.8	98.0	3.5	0.6	0.1
L9	60 °C	511	32.6	75.8	64.5	97.5	2.2	0.7	2.5
	100 °C	386	57.0	86.0	39.6	96.8	2.4	0.9	0.8
L10	60 °C	2136	45.2	86.9	50.5	96.8	3.8	0.5	1.2
	100 °C	2068	75.9	96.7	19.6	95.5	4.1	0.4	0.3
L11	60 °C	1064	42.8	84.3	53.1	95.1	3.6	0.5	1.4
	100 °C	1008	72.2	94.9	23.3	93.6	3.9	0.7	0.3
L12	60 °C	1899	47.3	88.0	47.4	95.9	3.8	1.5	1.1
	100 °C	1875	82.2	98.3	11.4	94.5	4.1	2.2	0.1
L13	60 °C	1818	40.8	87.9	55.3	98.5	3.8	0.1	1.5
	100 °C	1387	68.6	94.4	26.8	97.0	4.3	0.3	0.4
L14	60 °C	1803	49.8	86.8	46.6	95.6	3.5	0.1	1.0
	100 °C	1663	80.3	98.0	15.9	93.2	3.7	0.1	0.2
Chiraphos	60 °C	1805	36.1	89.1	60.3	98.6	3.4	0.2	1.8
	100 °C	1353	69.7	97.9	26.3	95.9	3.6	0.4	0.4
nBuPNP(o-F)	60 °C	354	22.1	82.0	74.6	97.5	2.3	1.0	4.0
	100 °C	335	48.9	92.2	47.6	95.6	2.4	1.2	1.0

[a] Standard conditions: 3 L autoclave, 1 L of MCH, 2 μmol of precatalyst, 500 equiv. of mMAO-3A, reaction time 1 h, 30 bar of ethylene pressure.

reaction. A screening test for the ethylene oligomerization was carried out at the 60 °C and 100 °C respectively, and the screening result was summarized in Table 1. A kinetic behavior was also measured by ethylene uptake profile (Fig. S2). Most of the *ortho*-fluorinated ligands showed similar tendency and no rapid decrease of ethylene uptake was observed at high temperature. It was observed that the productivity was increased when *ortho*-fluorine atom introduced to the near side from the bulkiness control substituent (L1 < L2, L5 < L6, L9〈L10), whereas fair-to-middling or slightly higher activity was shown to the far side (L1 < L3, L5≒L7, L9 < L11, L13≒L14). In the case of the *ortho*-fluorine atom was substituted to both sides, the activity was also effectively increased compared to the non-fluorine control group (L5 < L8, L9〈L12). In the case of L4, in which the smallest bulkiness alkyl and both side fluorine substituted, a significant amount of polymer was unexpectedly produced as a by-product. This phenomenon became worse as the temperature increased (9.2% at 60 °C, 32.0% at 100 °C). It is presumably due to the combined effects which are a steric effect of the substituted pendant fluorine atoms on both sides and an excessive free volume given by poor bulkiness. Some of the produced polymers were analyzed by GPC (Fig. S3) and DSC (Fig. S4). The molecular weight of L4, which had a higher polymer yield by side reaction, was in the range about 250,000 to 640,000, and the molecular weight distribution of the resulting polymer was considerably wide. Therefore presumed to be the main reason for the possibility of melting polymer by raised temperatures. The resulting polymer was also analyzed to HDPE (high-density polyethylene), which was generally crystalline, and this meant the product 1-hexene or 1-octene did not participate in the reaction. As the main feature in this system, it was observed that the activity was maintained with increasing reaction temperature in the experimental group in which the *ortho*-fluorine atom was substituted, while the activity systematically decreased in the control group (See the graphical data in Fig. S5 to comprehend the trend. Productivity drop on L1 by 25%, L5 by 30%, L9 by 24%, L13 by 24%, and Chiraphos by 25%). As a result of the experiment, it was considered that the bulkiest *t*-butyl substituent on the ethene backbone supposed to be the most efficient ligand system with an *ortho*-fluorine atom on the same side to the *t*-butyl substituent. The activity of this possible structure might be quite predictive; however, realistic evaluation of the target ligand could not be achieved because it was not synthesized in spite of many trials, probably due to the steric hindrance. Consequently, a trend of catalytic activity by changing substituent on the backbone tended to be increased by bulkiness (*n*-butyl≒phenyl < *i*-propyl < *t*-butyl). This was considered as a typical characteristic on the bidentate phosphine ligand series [21,27,28]. In case of introducing a bulky alkyl substituent, the movement and rotation of the neighboring aryl moiety would be limited, thus could affect on the reaction rate [28]. In the case of L6, which has a reduced bulkiness and *ortho*-fluorinated substituent at the same direction, it showed the best activity among the entries, and this activity was considered as the best performance among the existent PCCP ligand series. These results showed 2- to 7-fold greater activity based at 100 °C compared to other ligand systems (Chiraphos or *ortho*-fluorinated PNP). Therefore, it can be stated the newly developed catalyst series has an advantage in the production of selective LAOs (especially 1-hexene and 1-octene) showing relatively high activities at the high temperatures. It was noticeable that the product ratio for 1-octene *versus* 1-hexene tends to be somewhat lower than the entry of *ortho*-fluorinated PNP (4.0 for $^{o\text{-}F}$PNP, 1.3 for L6 at 60 °C, and 1.0 for $^{o\text{-}F}$PNP, 0.3 for L6 at 100 °C). It is corresponede that the small bite angle provides more tetramerization selectivity than trimerization selectivity. In general, the P-Cr-P bite angle in the PNP system is about 67^o, whereas the bite angle in the PCCP system is about 80^o [29]. Nevertheless, total obtained 1-octene yield produced by L6 was comparatively high enough to the conventional system.

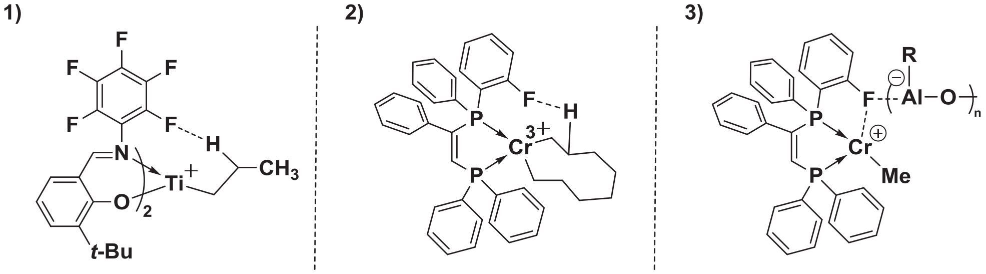


Fig. 1. 1) Weakly attractive and electrostatic interactions between the *ortho*-F and *β*-H in FI Catalyst, 2) in Chromium complex, 3) Alkyl-aluminum promotes catalytic activity with coordination of a fluoride atom.

3.2. Investigation into a role of the ortho-fluorine substituent

What the role of the *ortho*-fluorine atom is on the aryl phosphine to the metal center might be elucidated in analogous literatures. A stabilization effect to the metal center by the *ortho*-fluorinated phenyl substituent ligands was found in the functional FI catalysts known for the living-polymerization-based polymers [30–32]. It is reported that the FI catalyst is stabilized by coordination with an *ortho*-fluorinated substitution by noncovalent C-F···H-C interactions, and therefore monomers such as ethylene and *α*-olefins can be continuously inserted with a living character. (1 in Fig. 1) For this reason, the results of this study suggest that an *ortho*-fluorine atom stabilizes the intermediate; therefore ethylene is continuously introduced, and its activity maintains even at high temperatures. (2 in Fig. 1) Another possible suggestion can be found in “Fluoride effect” implying that an alkyl-aluminum promotes catalytic activity with coordination of a fluoride atom because of high electron affinity [33–35]. The bond enthalpies differ significantly (Al−F 664 kJ/mol, Cr−F 523 kJ/mol, and C−F 513 kJ/mol), suggesting that the resultant of Al···F bond interaction step should present a considerable driving force for a high ethylene incorporation. (3 in Fig. 1)

4. Conclusions

In conclusion, an *ortho*-fluorinated substitution was introduced to the conventional PCCP ligand system to overcome a limitation on using at high temperature. It was succeeded to synthesize newly manipulated ligand series and observed catalytic activities. The result shows an excellent performance which has not been recorded so far as high-temperature-operable oligomerization catalysts produced at the same pressure and reaction time. (*e.g.*, $(Ph^{o\text{-}F})(Ph)PC(i\text{-}pr) = C(iPr)PPh_2$ with an activity of 3246 kg/g Cr/h and selectivity of towards 74% of 1-hexene and 19% of 1-octene at 30 bar of ethylene, 100 °C in an hour of operation time) In this industry, this research is expected to contribute to finding a solution for operating selective oligomerization reaction continuously without any line plugging, with high activity simultaneously.

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Appendix A. Supplementary material

Crystal information files (cif), molecular structural data, and experimental details. See doi: https://doi.org/10.1016/j.catcom.2018.12.010 contains the supplementary crystallographic data for compound 1. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found in the online version, at https://doi.org/10.1016/j.catcom.2018.12.010.

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