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Note

Synthesis of novel polyfluorene with defined group in the center using aryl dipalladium complex as an initiator

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ABSTRACT

Dinuclear Pd(II) complex **DiPd–DPE** with a bridging ligand $C_6H_4OC_6H_4$-4,4′ was successfully synthesized by double oxidative addition reaction of $Pd(PCy_3)_4$ with 4,4′-dibromodiphenyl ether in good yield. X-ray crystal structure analysis reveals that the Pd atoms have the nearly square-planar coordination geometry with the similar bond lengths of two Pd–Br as well as four Pd–P, implying that two Pd(II) centers have similar reactivity. Using the dinuclear Pd(II) complex **DiPd–DPE** as an initiator, novel polyfluorene (PF) with a defined group ($C_6H_4OC_6H_4$-4,4′) in the middle of polymer main chain was prepared, demonstrated by MALDI-TOF analysis.

1. Introduction

Conjugated polymers have drawn a great deal of attention owing to their potential applications in organic light-emitting diodes (OLEDs) [1–8], field-effect transistors (OFETs) [9–11] and photovoltaic cells (OPVs) [12,13]. These polymers have been commonly synthesized by conventional step-growth polycondensation [14–16], displaying broad molecular weight distribution. Chain-growth polymerization, however, could address this issue and obtaining polymers with controlled molecular weight and low polydispersity [17–20]. The representative work involves the Ni-catalyzed polycondensation of Grignard-type monomer which was extensively used in the synthesis of regioregular poly(3-alkylthiopherens) (P3ATs) as an electron-donating material for OPVs [20–23]. The controlled regioregular polythiophenes with low polydispersity show superior electronic and optical properties over the regiorandom analogs. Recently, Yokozawa reported a three-coordinate complex $[(^tBu_3P)Pd(Ph)Br]$ used as an initiator in the chain-growth Suzuki–Miyaura polycondensation reaction of AB-type aromatic monomer. The polycondensation proceeded from the defined phenyl unit derived from the catalyst $[(^tBu_3P)Pd(Ph)Br]$ and achieved polymers with well-defined end groups [24–26]. Nevertheless, even with these advantages, the unsaturated-coordination catalyst $[(^tBu_3P)Pd(Ph)Br]$ has not been widely investigated in polymerization reaction due to its relatively poor stability, which must be strictly operated and stored under inert atmosphere at low temperature.

Inspired by these ideas, we speculate that if the polymerization starts from a saturated-coordination dinuclear palladium initiator with a bridging functional group between two Pd centers and proceeds in a chain-growth polymerization, the defined functional group would serve as an initiator unit and be incorporated in the center of polymer main chain. Therefore, we focus on the development of dipalladium catalyst [27–31], in which the choice of phosphine ligand is very critical. The bulky tricyclohexyl phosphine (PCy_3) was chosen as the auxiliary ligand because it has high activity in the reductive elimination step and could effectively inhibit the aryl–aryl exchange side reaction without the additional phosphine ligand [32–35]. In addition, diphenyl ether with simple structure and sufficient space is chosen as the bridging ligand. Finally, We successfully synthesized the dinuclear palladium complex **DiPd–DPE** by double oxidative addition of 4,4′-dibromodiphenyl ether to $Pd(PCy_3)_4$, and preliminarily investigate the polymerization behavior of AB-type fluorene monomer using **DiPd–DPE** as an initiator (Scheme 1).

2. Results and discussions

2.1. Synthesis of dipalladium complex **DiPd–DPE**

Based on the consideration of thermal stability, we initially attempted to prepare the dinuclear Pd(II) complex **DiPd–DPE** at

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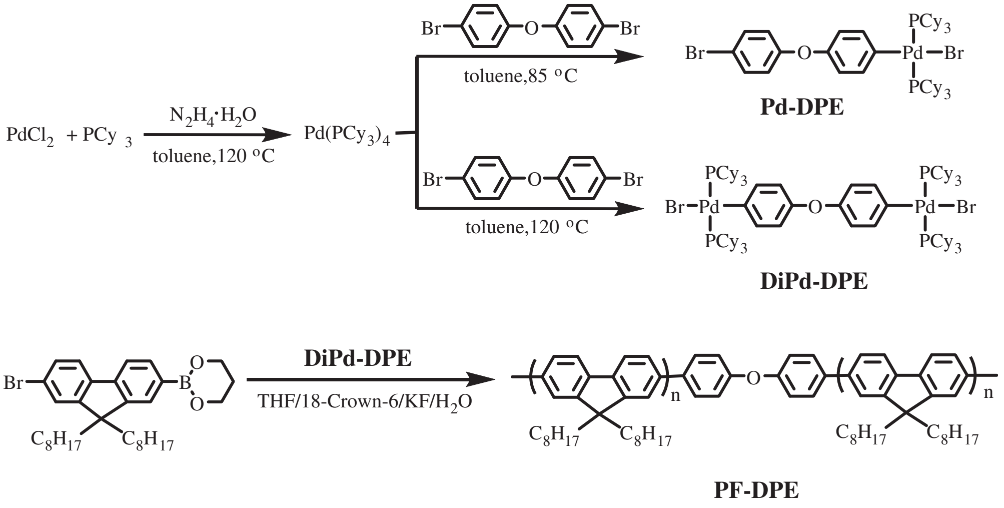

Scheme 1. Synthesis of dipalladium catalyst **DiPd–DPE** and polymer **PF–DPE**.

85 °C by double oxidative addition reaction of 4,4′-dibromodiphenyl ether with $Pd(PCy_3)_4$ generated *in situ* in a ratio of 1:2.5 in toluene, however, only mononuclear complex **Pd–DPE** was isolated in good yield, confirmed by ^1H NMR spectrum and X-ray crystallography. Increase of the reaction temperature to 120 °C leads to the target dipalladium complex **DiPd–DPE** in 72% yield, which means that the double oxidative addition could be carried out smoothly at higher temperature [24]. The chemical structure of **DiPd–DPE** was determined by ^1H NMR and ^{31}P NMR. ^{31}P NMR spectrum shows a singlet at 19.3 ppm, indicative of its symmetric structure, which is in accordance with a *trans* configuration having four magnetically equivalent PCy_3 ligands. The dipalladium(II) complex **DiPd–DPE** has better stability compared with monomeric three-coordination arylpalladium(II) halide complex $[(^tBu_3P)Pd(Ph)Br]$, which renders the operation easier. Indeed, complex **DiPd–DPE** can be weighted, transferred in air, and recrystallized from the solvents without distillation treatment. Furthermore, it is worth noting that the dipalladium(II) complex **DiPd–DPE** can be preserved in the Schlenk flask under argon at room temperature for two month with the catalytic activity unchanged.

2.2. X-ray characterization of **DiPd–DPE** and **Pd–DPE**

The molecular structures of complexes **DiPd–DPE** and **Pd–DPE** are shown in Figs. 1 and 2, and selected bond distances and angles were summarized in Table 1. Complex **DiPd–DPE** adopts the expected *trans* configuration with a nearly square-planar geometry around central Pd atoms, supported by the bond angle of C1–Pd1–Br1, P1–Pd1–P2, C10–Pd2–Br2 and P3–Pd2–P4 (166.15–173.80°), which are approximate to 180°. The four *cis* angles around each Pd atom are close to orthogonal (89.76–93.67° and 87.10–92.48°, respectively). The two planes defined by Pd1Br1P1P2C1 and Pd2Br2P3P4C10 are twisted with dihedral angle of 50.71°. Each phenyl ring is almost perpendicular to the adjacent Pd(II)-centered plane with the dihedral angles of 85.74 and 84.83°. In addition, the bond length of Pd1–Br1 [2.5324(7) Å] and Pd2–Br2 [2.5104(9) Å] are similar to each other as well as the four Pd–P bond distances. All of the results illustrate that two Pd(II) centers should have similar chemical reaction activity. Accordingly, the bridging group $C_6H_4OC_6H_4$-4,4′ would be introduced in the center of polymer main chain when the dipalladium(II) complex **DiPd–DPE** is used as an initiator in the polycondensation reaction.

2.3. Synthesis and polymerization behavior study of polyfluorene

The polymerization of AB-type monomer 2-(7-bromo-9,9-dioctylfluoren-2-yl)-1,3,2-dioxaborinane with dipalladium(II) complex **DiPd–DPE** was carried out at 25 or 75 °C for 12 h in the presence of KF/18-crown-6. THF with a small amount of water for dissolving KF was chosen as the reaction solvents because they could form a homogeneous system facilitating the polymerization process. After the reaction was quenched with 1 M hydrochloric acid, the crude product was filtered and washed with acetone via a Soxhlet apparatus to remove low-molecular-weight fractions. Molecular weights and polydispersity indices (PDI) of polyfluorene **PF–DPE** were determined by gel permeation chromatography (GPC) analysis with a polystyrene standard calibration. The number average molecular weight (M_n) of **PF–DPE** (25 °C) is 12,320 g/mol with a PDI of 1.84, and **PF–DPE** (75 °C) exhibits a higher M_n of 15,670 g/mol with a slightly broader PDI of 2.09. These results reveal that high temperature is beneficial for the proceeding of Suzuki–Miyaura coupling polymerization of fluorene-based monomer.

The polymerization behavior was further evaluated by MALDI-TOF mass spectrometry. The mass spectrum for polyfluorene obtained at 75 °C (Fig. 3) shows a series of major peaks accompanied with two sets of minor peaks. All of the mass differences between

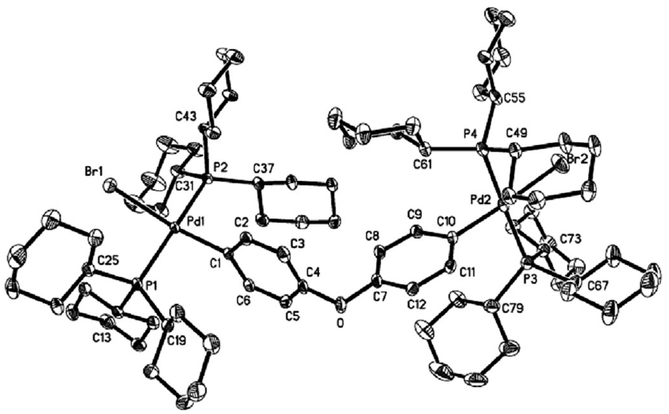

Fig. 1. An ORTEP drawing of **DiPd–DPE** showing the atom-labeling scheme and 30% probability thermal ellipsoids, H atoms and solvent molecule are omitted for clarity.

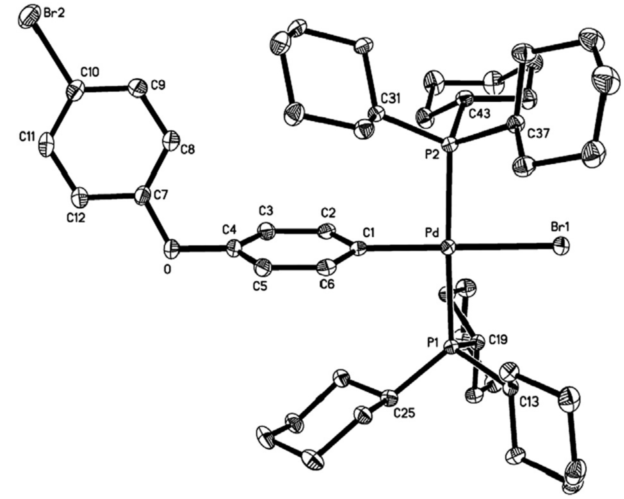

Fig. 2. An ORTEP drawing of **Pd**–**DPE** showing the atom-labeling scheme and 30% probability thermal ellipsoids, H atoms are omitted for clarity.

the adjacent main peaks are almost 388.6 such as 2502.0–2113.5 = 388.5, 2890.5–2502.0 = 388.5…, corresponding to the characteristic mass value of fluorene unit, which demonstrates that 9,9-dioctylfluorene is the repeating unit of afforded polymer. On the other hand, the major peaks correspond to polymer with diphenyl ether group ($C_6H_4OC_6H_4$-4,4′) in the center, derived from complex **DiPd**–**DPE**, and two hydrogen atoms at both ends, derived from the Pd complex end groups by quenching with hydrochloride acid (designed as DPE/H/H) [24–26,36]. For example, the mass of the 9-mer with DPE/H/H should be 3667.6 in theory and a signal peak at 3667.6 is observed in fact. The number of repeating unit for major signal peaks from left to right increases from 5 to 16. According to these results, we can conclude that the defined group ($C_6H_4OC_6H_4$-4,4′) is to a large degree incorporated into the middle of the polymeric main chain, implying that the polymerization at 75 °C mostly proceeds in a chain-growth manner.

The polymer obtained at 25 °C was also investigated by MALDI-TOF analysis. Unfortunately, the signal peaks containing the central $C_6H_4OC_6H_4$-4,4′ group could not be found in the mass spectrum. However, the repeating unit of the obtained polymer is still 9,9-dioctylfluorene unit, deducing from the mass differences between the adjacent peaks (388.6). The result indicates the polymerization behavior at low temperature (25 °C) is the traditional direct coupling of AB-type fluorene monomer rather than the typical chain-growth process. This behavior was also found in the polymerization carried out at 75 °C, deducing from two sets of minor peaks of the MALDI-TOF spectrum. According to the mechanism of the chain-growth polymerization, after the reduction elimination, the active Pd species should transfer and carry out the oxidative addition with the end C–Br, however, it is possible for the active Pd species to form free active ones. These species may in turn initiate the polymerization of monomer to give PF without **DPE** group. In spite of this, there exists a problem to speculate the precise molecule structures of these peaks based on their absolute mass because the corresponding *m*/*z* is not consistent with either the molecule weight of pure fluorene oligomers or the sum of central group $C_6H_4OC_6H_4$-4,4′ (168.2) plus any fluorene unit. Therefore, further work using the aryl Pd complexes with the functional group as initiator is in progress so as to discern reasonable supporting information.

Table 1
Selected bond lengths [Å] and angles [°] for **DiPd**–**DPE** and **Pd**–**DPE**.

Complex	DiPd–DPE			Pd–DPE	
Pd1–C1	2.008(5)	Pd2–C10	2.019(5)	Pd–C1	2.023(3)
Pd1–P1	2.3525(14)	Pd2–P3	2.3520(16)	Pd–P1	2.3681(7)
Pd1–P2	2.3687(14)	Pd2–P4	2.3637(15)	Pd–P2	2.3559(7)
Pd1–Br1	2.5324(7)	Pd2–Br2	2.5104(9)	Pd–Br1	2.5394(3)
C1–Pd1–P1	89.76(14)	P2–Pd1–Br1	87.10(4)	C1–Pd–P1	92.14(7)
C1–Pd1–P2	93.67(15)	C10–Pd2–P3	89.91(15)	C1–Pd–P2	90.64(7)
P1–Pd1–P2	169.60(5)	P3–Pd1–P4	171.42(5)	P1–Pd–P2	177.16(2)
C1–Pd1–Br1	166.15(16)	C10–Pd2–Br2	173.80(16)	C1–Pd–Br1	177.54(7)
P1–Pd1–Br1	91.91(4)	P3–Pd1–Br2	89.87(4)	P1–Pd–Br1	87.115(18)
P2–Pd1–Br1	87.10(4)	P4–Pd1–Br2	92.48(4)	P2–Pd–Br1	90.083(19)

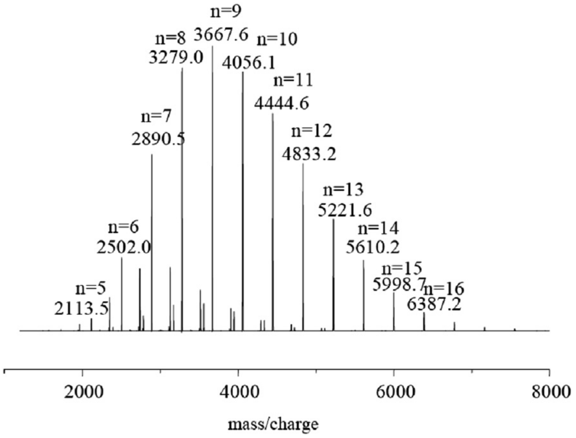

Fig. 3. MALDI-TOF mass spectrum of **PF**–**DPE** obtained at 75 °C.

3. Conclusions

In summary, we have successfully prepared a stable dinuclear palladium complex **DiPd**–**DPE** by double oxidative addition reaction of $Pd(PCy_3)_4$ generated *in situ* with 4,4′-dibromodiphenyl ether in good yield. Furthermore, a new-type conjugated polymer **PF**–**DPE** bearing diphenyl ether group in the center of polymer main chain was obtained using the complex **DiPd**–**DPE** as an initiator. The polymerization at high temperature starts from the diphenyl ether moiety derived from dipalladium complex **DiPd**–**DPE** and to a large extent proceeds in a chain-growth manner even with PCy_3 as auxiliary ligand. By reasonable modification of the bridging ligand of dinuclear palladium complex, we think, the polymers with the functional group in the center can be developed via the chain-growth polycondensation, and their photophysical properties might be tuned by the central group.

4. Experimental

4.1. General materials and measurements

All the reactions were carried out under argon atmosphere using standard Schlenk techniques. The solvents were dried and distilled under argon prior to use. All chemicals were purchased from the commercial sources such as Aldrich, J&K and Acros. ^1H NMR spectra with TMS as internal reference and ^{31}P NMR spectra with 85% H_3PO_4 as external reference were taken on a Bruker AV 400 NMR spectrometer at room temperature. Elemental analyses were performed on a BioRad elemental analysis system. Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) spectra were measured on an Autoflex III Bruker MALDI-TOF mass spectrometer.

4.2. Synthesis of dinuclear Pd(II) complex ***DiPd–DPE***

To a Schlenk flask containing $PdCl_2$ (0.44 g, 2.48 mmol) and PCy_3 (3.50 g, 12.48 mmol) at room temperature were added dry toluene (45 mL). The mixture was heated to 120 °C under argon and then Hydrazine hydrate (20 mL) was added dropwise to the solution. The mixture was stirred at 120 °C for 30 min to give a yellow solution. After cooled to the room temperature, the supernatant was transferred into a solution of 4,4′-dibromodiphenyl ether (0.33 g, 1.01 mmol) in toluene (10 mL) under Ar atmosphere. After stirring of the mixture at 120 °C for 24 h, the solvent was evaporated under reduced pressure. The residue was washed with Et_2O (3×10 mL) to give a white solid. The crude product was recrystallized from CH_2Cl_2/Et_2O to give **DiPd–DPE** (1.19 g, 72%) as colorless crystals. ^1H NMR (400 MHz, CD_2Cl_2, δ/ppm): 7.18 (d, $J = 7.9$ Hz, 4H, phenylene), 6.58 (d, $J = 7.9$ Hz, 4H, phenylene), 2.07–1.93 (m, 12H, PCy_3), 1.85–1.77 (m, 24H, PCy_3), 1.68–1.47 (m, 60H, PCy_3), 1.18–0.97 (m, 36H, PCy_3). $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$, δ/ppm): 19.3 (s). Anal. Calcd. for $C_{84}H_{140}Br_2OP_4Pd_2 \cdot Et_2O$: C, 60.86; H, 8.71. Found: C, 60.71; H, 8.70.

Synthesis of mononuclear Pd(II) complex ***Pd–DPE***: The same procedure as the preparation of **DiPd–DPE** was used but at 85 °C rather than 120 °C. Yield 0.64 g, 64%. ^1H NMR (400 MHz, $CDCl_3$, δ/ppm): 7.37 (d, $J = 8.4$ Hz, 2H, phenylene), 7.34 (d, $J = 8.8$ Hz, 2H, phenylene), 6.85 (d, $J = 8.8$ Hz, 2H, phenylene), 6.71 (d, $J = 8.4$ Hz, 2H, phenylene), 2.08–1.90 (m, 20H, PCy_3), 1.76–1.54 (m, 28H, PCy_3), 1.25–1.06 (m, 18H, PCy_3). Anal. Calcd for $C_{48}H_{74}Br_2OP_2Pd$: C, 57.92; H, 7.49. Found: C, 58.22; H, 7.54.

4.3. Synthesis of polyfluorene ***PF–DPE***

To a Schlenk flask containing the monomer 2-(7-bromo-9,9-dioctylfluoren-2-yl)-1,3,2-dioxaborinane (0.28 g, 0.51 mmol), 18-crown-6 (0.53 g, 2.01 mmol), KF (0.12 g, 2.01 mmol) were added a mixture of THF/H_2O (8 mL/1 mL) in that order. To another Schlenk containing dinuclear Pd(II) complexes **DiPd–DPE** (33.3 mg, 0.02 mmol) were added THF (7 mL). The solution of **DiPd–DPE** was transferred into the solution of monomer in THF/H_2O. The reaction mixture was stirred at 25 or 75 °C for 12 h. The resulting mixture was cooled to room temperature, and then poured into dichloromethane (200 mL). The dilute aqueous HCl (10 mL, 1 M) was added. After stirring at room temperature for 30 min, the mixture was washed with water and the organic layer was dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure. The residue was precipitated from dichloromethane/MeOH to give the crude polymer. Then the solid was loaded into Soxhlet apparatus and washed with acetone (12 h). The remaining polymer was dried over in a high vacuum drying oven overnight. Yield: 86%.

4.4. X-ray structure determination of ***DiPd–DPE***

Crystals of **DiPd–DPE** and **Pd–DPE** suitable for X-ray crystallography were obtained by recrystallization from dichloromethane/diethyl ether. X-ray data were collected on a Bruker Smart APEX diffractometer equipped with CCD detector and graphite monochromator, Mo Kα radiation ($\lambda = 0.71073$ Å). The intensity data were recorded with ω scan mode (187 K). Lorentz, polarization factors were made for the intensity data and absorption corrections were performed using SADABS program [37]. The crystal structure was determined using the SHELXTL program and refined using full matrix least squares [38]. All non-hydrogen atoms were assigned with anisotropic displacement parameters, whereas hydrogen atoms were placed at calculated positions theoretically and included in the final cycles of refinement in a riding model along with attached carbons.

Acknowledgments

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Appendix A. Supplementary material

CCDC 923881 and 923882 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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