Synthesis of Conjugated Copolymer Containing Spirobifluorene Skeleton by Acyclic Diene Metathesis Polymerization for Polymer Light-Emitting Diode Applications

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Grubbs-type, Hoveyda-type, cyclic alkyl amino carbene (CAAC)-based ruthenium olefin metathesis catalysts were used for the acyclic diene metathesis (ADMET) polymerization of 2,7-divinyl-9,9-di-*n*octylfluorene (DVF). Additionally, various ratios of DVF and 2,2',7,7'-tetravinyl-9,9'-spirobifluorene (TVSF) were subjected to ADMET polymerization to obtain polymers P1–P7. Polymers P1–P4 were analyzed with gel permeation chromatography, UV–vis spectroscopy, and photoluminescence spectroscopy. As the TVSF ratio increases, polymers exhibit lower solubility but a narrower band in the photoluminescence spectrum. Polymer light-emitting diode (PLED) devices were fabricated with polymers P1, P2, and P3. The performances of the PLED devices indicated that polymers including more spirobifluorene blocks showed better turn-on voltage, brightness, current efficiency, and power efficiency.

Keywords: Polymer, Polymer organic light-emitting diodes, Acyclic diene metathesis polymerization, Copolymer, Spirobifluorene

Introduction

Conjugated polymers have been evolving since their discovery to produce light-weight, low-cost, and large-area electronic devices such as organic solar cells,¹ organic thinfilm transistors,² and organic light-emitting diodes.³ The optical, electrical, and morphological properties of conjugated polymers can be tuned easily by adjusting the type of monomer backbone and the side chain combination. Conjugated polymers containing vinyl groups are one type that has been researched.⁴ The vinyl group in conjugated polymers is the simplest unit block that can extend the conjugation length and act as a spacer between the two units, affecting coplanarity and interchain interactions. To introduce vinyl groups in conjugated polymers, Gilch polymerization,⁵ Heck coupling reaction,⁶ and Horner– Emmons reactions⁷ have been used.

Additionally, the olefin metathesis reaction allows the formation of carbon–carbon double bonds by the redistribution of olefins.⁸ Highly reactive, stable, selective ruthenium metathesis catalysts have been developed⁹ for various types of reactions, such as cross metathesis (CM), acyclic diene metathesis (ADMET), ring-closing metathesis, ring-opening

metathesis, ring-opening metathesis polymerization, and ethenolysis.¹⁰

We noticed that compared to other reactions for the synthesis of conjugated polymers, ADMET polymerization affords no deleterious impurities and no defective polymers, and it is a reversible reaction.¹¹ Thus, conjugated polymers synthesized through ADMET polymerization would have structural regularity and chemical purity, which could be beneficial for application to optoelectronic and electrochemical devices. Therefore, ADMET polymerization to synthesize conjugated polymers has been studied.¹² Nevertheless, to the best of our knowledge, backboneconjugated polymers polymerized by metathesis reactions have not been applied to polymer light-emitting diode (PLED) devices. We envisioned that synthesizing conjugated polymers with ADMET polymerization could be appropriate for fabricating PLEDs. We selected 2,7-divinyl-9,9-di-n-octylfluorene (DVF) and 2,2',7,7'-tetravinyl-9,9'spirobifluorene (TVSF) as monomers, which showed good performance in PLEDs and stable photoluminescence spectra, respectively.¹³

Herein, we describe the activity of various ruthenium catalysts^{9,14} for ADMET polymerization, the synthesis of copolymers by ADMET copolymerization with DVF and

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TVSF, and the physical and optical properties of these copolymers as determined by ¹H nuclear magnetic resonance (NMR), gel permeation chromatography (GPC), UV–vis spectroscopy, and photoluminescence spectroscopy. Additionally, PLEDs were fabricated from the copolymers, and the performances of the devices were confirmed.

Results and Discussion

DVF and ruthenium metathesis catalysts were synthesized by a previously reported method.^{6a,15} To determine the ADMET polymerization reactivity of each type of ruthenium catalyst, ADMET polymerization was performed with ruthenium catalysts **1–8** (Table 1). Among Ru catalysts **1– 8**, the ADMET polymerization of DVF with catalyst **1** afforded the highest degree of polymerization of 40 under the given reaction conditions. Relatively low degrees of polymerization (DPs) 13 and 29 were attained with **2** and **3**, respectively. ADMET polymerization with Z-selective catalyst **4** afforded no polymer.

Interestingly, the activities of cyclic alkyl amino carbene (CAAC)-liganded ruthenium complexes strongly correlated

with N-aryl substitution. With *N*-2,6-diethyl-phenyl-bearing CAAC, ruthenium catalysts **5** and **6** exhibited moderate activity with yield of 68% on ADMET polymerization, while *N*-2,6-diisopropyl-phenyl-bearing CAAC ruthenium catalysts **7** and **8** afforded no polymers in ADMET polymerization. *N*-2,6-diisopropyl-phenyl-bearing CAAC ruthenium catalysts showed suitable reactivity in general ring-closing metathesis, CM, and ethenolysis,^{14,15} but not in ADMET polymerization of DVF. These results suggest that the *N*-2,6-diisopropyl-phenyl group in CAAC is not sterically compatible with the metathesis reaction with DVF.

Not only the N-aryl group in CAAC but also the substituent at the quaternary carbon in CAAC ruthenium catalysts is related to ADMET polymerization activity. The phenyl ring instead of a methyl group on the quaternary carbon atom of the CAAC ruthenium catalyst results in a higher DP 14 for **5** vs. DP 22 for **6**. These results are consistent with the previously reported tendencies of CAAC-based Ru catalysts for other metathesis reactions.^{14,15a,15b}

TVSF was synthesized by the Stille coupling reaction of 2,2',7,7'-tetrabromo-9,9'-spirobifluorene by a previously

Table 1. Acyclic diene metathesis polymerization of 2,7-divinyl-9,9-dialkylfluorenes with various ruthenium catalysts.



Catalyst	$M_{\rm n}^{\rm a}$ (g mol ⁻¹)	$M_{\rm w}^{\rm a}$ (g mol ⁻¹)	PDI ^a	Yield ^b	DP ^c
1	12 000	20 000	1.6	73%	40
2	3200	6700	2.0	70%	13
3	5200	15 000	2.8	74%	29
4	_	_	-	d	_
5	2900	4800	1.7	68%	14
6	3800	8000	2.1	68%	22
7	_	_	_	-	_
8	-	-	-	-	-

^a GPC data.

^b Isolated yield.

^c The degree of polymerization (DP) was estimated based on the integration ratios in ¹H NMR spectrum.

^d Nothing precipitated in methanol.

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Table 2. Acyclic diene metathesis (ADMET) copolymerizations of 2,7-divinyl-9,9-dialkylfluorenes (DVF) and 2,2',7,7'-tetravinyl-9,9'-spirobifluorene (TVSF).



	DVF:		$M_{\rm n}^{\rm b}$	$M_{\rm w}^{\rm b}$	
Polymer	TVSF	Yield ^a	$(g \text{ mol}^{-1})$	$(g \text{ mol}^{-1})$	PDI
P1	1:0	73%	12 000	20 000	1.6
P2	20:1	75%	7600	19 000	2.4
P3	10:1	71%	6700	14 000	2.0
P4	5:1	76%	4600	8700	1.8
P5	3:1	86%	_	_	-
P6	1:1	96%	_	_	-
P7	0:1	97%	_	-	-

^a Isolated yield.

^b GPC data.

reported method.^{11h} The ADMET copolymerization experiments were performed using different DVF/TVSF ratios (1:0 to 0:1). TVSF can work as a crosslinking agent, and, in fact, many portions of the generated polymers were insoluble in any organic solvents especially at high TVSF/ DVF ratios.

Since appropriate solubility is required for device fabrication, soluble polymers with four polymerization block ratios (DVF:TVSF = 1:0, 20:1, 10:1, 5:1) were analyzed by GPC (Table 2). As the TVSF/DVF ratio increases, the $M_{\rm p}$ and $M_{\rm w}$ of the polymer decreased; crosslinked or high-molecularweight polymer chains were possibly discarded in GPC measurement, leaving only low-molecular-weight polymer chains for the batches performed at a TVSF/DVF ratio.

To evaluate the DVF/TVSF ratio-dependent optical properties of P1-P4, UV-vis spectroscopy and photoluminescence spectra of fabricated films were obtained.

Figure 1(a) shows the UV-vis absorption spectrum of the four polymer films (P1-P4) prepared by spin casting on glass. The spectrum for P1 shows two absorption bands at



Figure 1. UV-vis and photoluminescence spectra of P1-P4.

Table 3. PLED device performance of P1–P3.									
Polymer	Thickness (nm)	Turn- on voltage (V)	Maximum luminance (cd/m ²)	Current efficiency (cd/A)	Maximum power efficiency (Im/W)				
P1	115	10	21	0.01	0.003				
	95	8	25	0.01	0.003				
P2	75	9	113	0.03	0.01				
	65	7	78	0.03	0.01				
P3	95	8	161	0.05	0.01				
	75	8	169	0.03	0.01				

428 and 461 nm, which come from π - π * transitions of the conjugated backbone. As the ratio of TVSF in the polymer increased (P2, P3, and P4), the 461 nm band absorption decreased and became slightly redshifted compared to that of homopolymer P1. The difference might result from the increase in the TVSF ratio, which decreases the degree of polymerization and shortens the conjugation length. Figure 1(b) shows the photoluminescence spectra of the four polymer films (P1-P4) prepared by spin casting on glass. The spectra for P1 to P4 all showed a strong emission band at 465 nm with two shoulder bands at 496 and 530 nm. The results indicate that the replacement of the fluorene unit with a spiro-connected bifluorene does not change the conjugated backbone electronic properties of polymers. However, when comparing P1 to P3, as the ratio of TVSF increases, the shoulder peak (496 nm, 530 nm) of photoluminescence decreases. P3, which contains a high TVSF ratio, has decreased interchain interactions due to steric hindrance of the spiro group, thus emitting a narrower, purer blue color in the photoluminescence spectrum. Exceptionally, P4 has a broad spectrum due to the low degree of polymerization of the polymer, which comes from a high ratio of insoluble comonomer TVSF.

To confirm the effect of the spirofluorene structure in PLED devices, ITO/PEDOT:PSS/polymer/Ca/Al was fabricated with soluble polymers P1, P2, and P3. The electroluminescence performances of the three copolymers are listed in Table 3. Compared with homopolymer P1, copolymers P2 and P3 show slightly lower turn-on voltages, which is better for device operation. Additionally, the brightness, current efficiency, and power efficiency of the device improved in the order of P1, P2, and P3, respectively. This result means that increasing the incorporation ratio of the spirobifluorene building block improved the PLED device performances. Effect of TVSF on photophysics and device performance might be attributed to the difference in the shape of polymer chain. Without TVSF, linear polymer chains are generated and stacking of chains could occur. By feeding TVSF, branched polymers are produced and chain stacking might be blocked.

We compared these PLED device performances with reported poly(fluorenevinylene)-based PLEDs.5a,16 PLED

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device performances with Gilch and Horner-Emmons reaction polymers showed lower turn-on voltage (3-4 V), higher brightness (870 cd/m^2), and higher current efficiency (0.16-6.5 cd/A) than those of ADMET reaction polymers P1-P3. The PLED device performance could be correlated with the molecular weight of the polymer used in the device fabrication. The molecular weights of the polymers synthesized by Gilch and Horner-Emmons methods are higher (Mw = $222\ 000$ and $78\ 000$, respectively) than those of the P1-P3 polymers by ADMET. ADMET polymers have lower molecular weights, more chain ends, and higher chain mobility, which can lead to the formation of more excimers and aggregates, resulting in lower PLED performance.¹⁷ Therefore, to improve the PLED performances of ADMET polymers, more efficient metathesis catalysts affording ADMET polymers with higher polymerization degrees should be developed.

Conclusion

We have synthesized fluorene-divinylene-based polymers by ADMET polymerization with Grubbs-type, Hoveydatype, CAAC-based ruthenium olefin metathesis catalysts. Under optimized catalysts and conditions, we copolymerized various ratios of DVF/TVSF by ADMET polymerization. The properties of the synthesized copolymers P1 to P7 and the GPC results indicate that a higher ratio of TVSF during copolymer polymerization leads to lower solubility and thus decreases the degree of polymerization. Investigation by UV-vis spectroscopy showed that an increase in the TVSF ratio in the copolymer did not change the electronic properties of the backbone, but showed a narrow band in the photoluminescence spectrum. PLED devices were fabricated with P1, P2, and P3, and the results indicated that the more spirobifluorene blocks were included, the better was the performance of the device.

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Conflict of Interest. The authors declare no conflict of interest.

Supporting Information. Additional supporting information may be found online in the Supporting Information section at the end of the article.

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