

Synthesis of Polyphenylenes through Bergman Cyclization of Eneidyne^{*} with Long Chain Alkyl Groups^{*}

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Abstract Several new eneidyne with long chain alkyl groups were synthesized through Sonogashira coupling reactions between long chain alkynes and (Z)-1,2-dichloroethene. These eneidyne (1) were then subjected to thermal Bergman cyclization in a refluxing diphenyl ether bath under vacuum to obtain conjugated polyphenylenes with the weight-average molecular weights up to $4.9 \times 10^3 \text{ g}\cdot\text{mol}^{-1}$. The occurrence of Bergman cyclization was confirmed by ¹H-NMR, ¹³C-NMR, and IR spectroscopic analysis. These polyphenylenes are fully soluble in common organic solvents and exhibit good thermal stability. The optical properties of the polyphenylenes were investigated by UV-Vis absorption and photoluminescence (PL) spectroscopies. A blue emission was observed for all these polyphenylenes.

Keywords: Eneidyne; Bergman cyclization; Polyphenylene.

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INTRODUCTION

Conjugated polymers are a class of polymers with unique thermal, optical and electrical properties^[1–9]. Several families of conjugated polymers, including poly(*p*-phenylene) (PPP), polyacetylene (PA), poly(phenylenevinylene) (PPV), poly(phenyleneethynylene) (PPE), polypyrrole (PPy) and polythiophene (PT), have been well developed after the introduction of the concept of conducting polymers in the 1970s. Most of the conjugated polymers were prepared by transition-metal-catalyzed cross-coupling reaction^[10–12]. The transition metal catalyst however may seriously affect the electrical properties of the conjugated polymers as the residual metal prefers to weakly coordinate with the conjugated backbone^[13], leaving a great challenge to the purification of the polymers, which may hinder the applications of conjugated polymers in diverse area of applications such as batteries, capacitors, light-emitting diodes and transistors. Developing metal-free synthesis of conjugated polymers is therefore of great research interests.

Poly(*p*-phenylenes) (PPPs) contain unique structural features compared with other conjugated polymers, which make them a promising class of organic materials since they combine excellent thermal and mechanical properties of the rigid aromatic macromolecules with attractive optoelectronic properties of the semi-conducting

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polymers. In addition to displaying liquid crystallinity^[14] and nonlinear optical properties, these materials are currently used as active components in several electronic devices, solar cells, electrode materials for chemical power sources, and essentially in polymeric light emitting diodes (PLEDs)^[15–19]. PPPs have been synthesized through different techniques including oxidative coupling, Yamamoto coupling reaction^[20], Suzuki coupling reaction^[21], Kumada coupling reaction^[22] and electrochemical polymerization^[23]. The structural viability, however, is limited due to the nature of these coupling reactions. Herein, we wish to report our work by using Bergman cyclization polymerization to construct PPPs.

Bergman cyclization is a cycloaromatization of enediyne compounds, leading to the formation of 1,4-phenylene biradicals^[24]. The multi-stimuli-triggered nature of this reaction and the high reactivity of the biradical intermediates generated from this reaction have attracted much research attention^[25–31] since the first report by Bergman *et al.*^[24]. Besides being used in pharmaceutical research and organic synthesis due to the cytotoxicity of the biradical intermediate^[32], Bergman cyclization has also been widely used as a promising tool for polymer chemistry and material science as they can be triggered without transition metal catalyst^[33]. We noticed that PPPs were synthesized from Bergman cyclization of dialkynylethylene^[34], while some of the resultant materials were predominantly the unreacted monomer. In addition, because of the high rigidity and crystallinity of the backbone, most PPPs were insoluble, intractable, and infusible, hindering their further application in processing and device integration. In this work, we introduced long alkyl chains to enediyne molecules to overcome the solubility problem of final PPPs. After the thermal triggered Bergman cyclization at a particular temperature revealed by differential scanning calorimetry (DSC), soluble PPPs were obtained. The backbone structure and properties of the conjugated polymers were characterized on the basis of a variety of analytic techniques.

EXPERIMENTAL

Materials

n-Butylamine and *N,N*-dimethylformamide (DMF) were dried over calciumhydride (CaH₂) and then distilled. 1-Bromoheptane, 1-bromooctane, 1-bromononane, 1-bromodecane, 1-bromohexadecane, 1-bromooctadecane were purchased from Aladdin Industrial Corporation. (Z)-1,2-Dichloroethene was obtained from TCI Chemical Reagent Co., Ltd. Tetrabutylammonium fluoride (TBAF) and trimethylsilylacetylene (TMSA) were obtained from Shanghai Darui Finechemical Co., Ltd. Other reagents used in the experiments were obtained from commercial sources as reagent grade and used as received without further purification. All the Sonogashira reactions were performed with dry Schlenk techniques under an atmosphere of nitrogen unless otherwise noted. All the Bergman cyclization reactions were performed under vacuum in a sealed glass tube with refluxing fluid outside as heating medium. Trimethylsilylacetylene derivatives and corresponding deprotected long chain alkynes are known compounds, and were synthesized according to literature procedures with minor modifications^[35, 36].

Characterization

¹H-NMR (400 MHz) and ¹³C-NMR (100 MHz) spectra were recorded in Chloroform-*d* (CDCl₃) on an Ultra Shield 400 spectrometer (BRUKER BIOSPIN AG, Magnet System 400 MHz/54 mm). Chemical shifts were reported downfield from the Me₄Si resonance which was used as the internal standard. Fourier transform infrared spectra (FTIR) were recorded from KBr pellets on a Nicolet Magna 5700 FTIR spectrometer. The differential scanning calorimetry (DSC) was carried out on a SPA409PC Thermal Analyzer. The samples were heated at a constant rate of 10 K/min from room temperature to 350 °C under N₂ flow. Thermogravimetric analysis (TGA) was carried out at a heating rate of 10 K/min under nitrogen using a Netzsch STA 449F3 thermogravimetric analyser. The weight-averaged molecular weight (*M*_w) and the polydispersity index (PDI) of the polymers were determined by gel permeation chromatography (GPC) on a WATERS 1515 equipped with a series of PS gel columns, using THF as an eluent at 40 °C with a PS calibration. Absorption spectra were recorded on a Shimadzu UV-2550 UV-Vis spectrometer. The steady-state fluorescence experiments were performed on a Varian Cary Eclipses fluorescence spectrometer. Mass spectra (MS) were recorded on a Micromass LCTTM mass spectrometer using EI method.

Synthesis of Enediyne Compounds (1)

(Z)-1,2-Di(1-nonynyl)ethene (**1a**): CuI (675.8 mg, 0.4 mmol, 10%), Pd(PPh₃)₂Cl₂ (140.4 mg, 0.2 mmol, 10%) were added in a 50 mL Schlenk flask. Then (Z)-1,2-dichloroethene (188.97 mg, 2 mmol), DMF (15 mL) and *n*-BuNH₂ (2 mL) were added successively under a nitrogen atmosphere. 1-Nonyne (745.32 mg, 6 mmol) was added after the flask was subjected to three freeze-pump-thaw cycles. The reaction mixture was stirred at 40 °C for 4 h. The resulting brown solution was partitioned with saturated brine solution and dichloromethane, and the organic layer was dried over anhydrous MgSO₄. After removal of the solvent under vacuum, the crude product was purified through alumina column chromatography (petroleum ether) to yield the title compound as a yellow liquid oil (0.354 g, 65%). ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.60–1.53 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.46–1.37 (m, CH_3-CH_2- , 4H), 1.34–1.28 (m, $-\text{CH}_2-$, 12H), 0.90–0.86 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 97.8, 78.2, 31.8, 28.9, 28.8, 28.7, 22.6, 19.8, 14.0. HR-MS: *m/z* calcd. for C₂₀H₃₂: 272.2504; found: 272.2506.

All other enediyne were synthesized in a similar manner.

(Z)-1,2-Di(1-decynyl)ethene (**1b**)^[34]: Yellow oil, 70%. ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.59–1.53 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.45–1.40 (m, CH_3-CH_2- , 4H), 1.29–1.27 (m, $-\text{CH}_2-$, 16H), 0.90–0.87 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 97.9, 78.3, 31.9, 29.2, 29.1, 28.9, 28.7, 22.7, 19.8, 14.1.

(Z)-1,2-Di(1-undecynyl)ethene (**1c**): Yellow oil, 68%. ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.60–1.52 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.44–1.38 (m, CH_3-CH_2- , 4H), 1.38–1.27 (m, $-\text{CH}_2-$, 20H), 0.89–0.86 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 97.9, 78.2, 31.9, 29.5, 29.3, 29.2, 28.9, 28.7, 22.7, 19.8, 14.1. HR-MS: *m/z* calcd. for C₂₄H₄₀: 328.3130; found: 328.3132.

(Z)-1,2-Di(1-dodecynyl)ethene (**1d**): Colorless oil, 62%. ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.60–1.52 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.44–1.38 (m, CH_3-CH_2- , 4H), 1.38–1.26 (m, $-\text{CH}_2-$, 24H), 0.89–0.86 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 98.0, 78.2, 31.9, 29.6, 29.6, 29.3, 29.2, 28.9, 28.7, 22.7, 19.8, 14.1. HR-MS *m/z* calcd. for C₂₆H₄₄: 356.3443; found: 356.3444.

(Z)-1,2-Di(1-octadecynyl)ethene (**1e**): White solid, 75%. ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.60–1.52 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.44–1.40 (m, CH_3-CH_2- , 4H), 1.37–1.26 (m, $-\text{CH}_2-$, 48H), 0.90–0.86 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 97.9, 78.3, 32.0, 29.7, 29.7, 29.6, 29.4, 29.2, 28.9, 28.7, 22.7, 19.8, 14.1. HR-MS: (M)⁺ *m/z* calcd. for C₃₈H₆₈: 524.5321; found: 524.5323.

(Z)-1,2-Di(1-icosynyl)ethene (**1f**): White solid, 72%. ¹H-NMR (CDCl₃, 400 MHz, δ): 5.72 (s, $-\text{HC}=\text{CH}-$, 2H), 2.40–2.37 (s, $-\text{C}\equiv\text{C}-\text{CH}_2-$, 4H), 1.60–1.53 (m, $-\text{C}\equiv\text{C}-\text{CH}_2-\text{CH}_2-$, 4H), 1.44–1.40 (m, CH_3-CH_2- , 4H), 1.26 (m, $-\text{CH}_2-$, 56H), 0.90–0.87 (t, $-\text{CH}_3$, 6H). ¹³C-NMR (CDCl₃, 100 MHz, δ): 118.9, 97.9, 78.2, 31.9, 29.7, 29.7, 29.6, 29.4, 29.3, 28.9, 28.7, 22.7, 19.8, 14.1. HR-MS: *m/z* calcd. for C₄₂H₇₆: 580.5947; found: 580.5948.

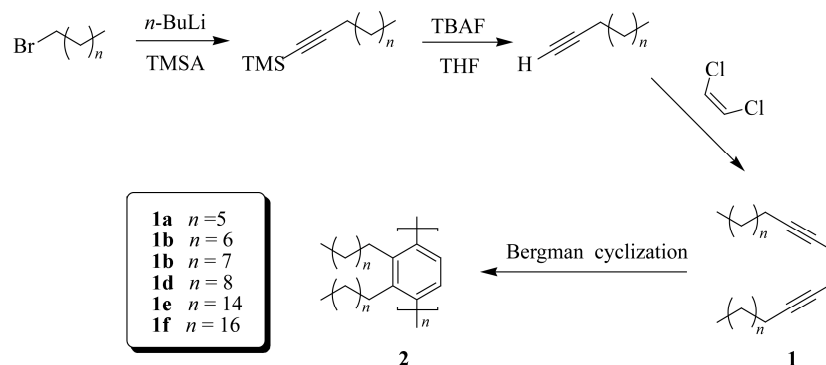
General Procedure for the Synthesis of Polyphenylenes (2)

Enediyne **1** was added into a tube and sealed under vacuum. The tube was then embedded in a refluxing diphenyl ether bath. After 8 h of heating, the polyphenylene was obtained as a dark-brown oil, which is soluble in toluene, DCM, THF, DMF *etc.*

RESULTS AND DISCUSSION

Scheme 1 shows the synthetic procedure of the polyphenylenes with long alkyl side chains. Treating TMSA with alkylbromide in the presence of *n*-BuLi gave the protected long chain alkynes, which were subjected to the deprotection to form long chain alkynes in the presence of TBAF in THF in a quantitative yield. The Sonogashira coupling reaction between long chain alkynes and (Z)-1,2-dichloroethene in the presence of

palladium catalyst gave the desired enediyne monomers **1** in good to high yield. The structures of all these compounds were confirmed by ^1H - and ^{13}C -NMR and high resolution MS analysis.



Scheme 1 Synthesis of long alkyl-based enediyne compounds and Bergman cyclization

The thermal triggered Bergman cyclization of enediyne compounds were visualized with DSC. Figure 1 shows the exothermic peaks for enediyne **1d** and **1f** appearing at 234 °C and 200 °C respectively, which correspond to the occurrence of Bergman cyclization of the enediyne moiety. It should be noted that in addition to the exothermic peak, a strong sharp endothermic peak at 53 °C was observed for enediyne **1f**, which can be ascribed to the melting of the crystals. Taking into account the alkyl chain lengths of the other enediyne compounds are similar to that of **1d** and **1f**, we deduced that the onset temperature of Bergman cyclization for the other enediyne compounds are in the range of 200–250 °C, which makes it a reasonable choice for us to choose diphenyl ether as a heating medium.

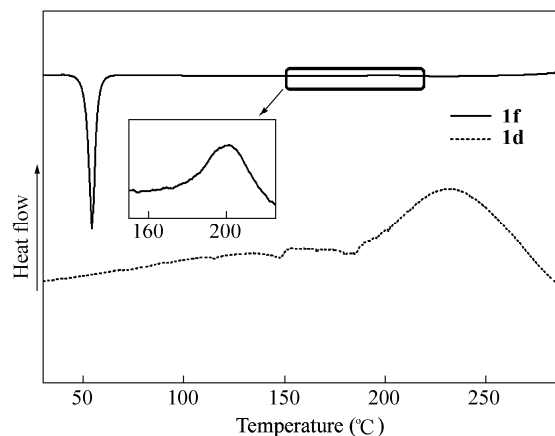


Fig. 1 DSC curves of representative enediyne compounds **1d** and **1f** (The inset shows the enlarged exothermal peak of compound **1f**.)

Thermal-triggered Bergman cyclization polymerization of these enediyne compounds was conducted under vacuum to eliminate any species that can quench radicals. After heating in a bath of boiling diphenyl ether (259 °C) for 8 h, brown viscous oils for **2a–2f** were obtained in a quantitative yield. Gel permeation chromatography (GPC) showed narrow molecular weight distributions (Table 1). The weight average molecular weights estimated through GPC using polystyrene standards were between 2000 and 5000, the corresponding degrees of polymerization (DP) was thus estimated to be between 7 and 11. Both molecular weights and DPs of **2a–2f** are higher than that of polyphenylenes generated from maleimide-based enediyne^[37], which is due to the relatively small steric hindrance of the long alkyl chains than aryl rings. It should be noted that these oligomers

have more compact structures and smaller hydrodynamic volumes compared with the polystyrene standard. The molecular weights and DPs should thus be higher than the values obtained from GPC analysis. Nevertheless, the DPs of the polymers are comparable to the effective conjugation length^[2] of other PPPs to be useful for luminescence applications.

Table 1. Weight-average molecular weights and PDI of the obtained PPPs^a

	2a	2b	2c	2d	2e	2f
M_w	2200	2400	3200	4200	4700	4900
PDI	1.41	1.26	1.50	1.60	1.30	1.28

^a Molecular weights and PDI were obtained using GPC with THF as eluent and polystyrene as standard

FTIR spectroscopy analysis was employed to confirm the occurrence of Bergman cyclization. Figure 2 shows a comparison of IR spectra of the representative enediyne precursor (**1d**) and the corresponding PPP (**2d**). The stretching band of $C\equiv C$ at 2202 cm^{-1} for **1d** disappears for **2d**, which directly evidences the reaction of the alkynyl groups after heating. The formation of the conjugated polyphenylenes through Bergman cyclization was also confirmed by NMR spectroscopy. Figure 3 shows the ^1H - and ^{13}C -NMR spectra of a representative enediyne compound **1d** and the corresponding polymer **2d**. The single peak associated with the vinyl protons at $\delta = 5.72$ is completely disappeared after the polymerization. Meanwhile the peaks associated with the methylene protons adjacent to carbon-carbon triple bond at $\delta = 2.37\text{--}2.40$, the methylene protons adjacent to methyl group at $\delta = 1.38\text{--}1.43$, the rest methylene protons at $\delta = 1.26\text{--}1.60$ and the methyl protons at $\delta = 0.86\text{--}0.89$ are all broadened after the polymerization, which is a direct evidence for the formation of a rigid polymer backbone. The occurrence of Bergman cyclization polymerization could also be revealed *via* ^{13}C -NMR analysis. After the Bergman cyclization, both characteristic peaks of the alkynyl group at $\delta = 78$ and 98 and the vinyl group at $\delta = 119$ were completely gone, whereas peaks at $\delta = 14\text{--}32$ corresponding to the methylene carbons and methyl carbons are broadened, indicative of the formation of the polymer. Note that there is no peak appears in the aromatic region, which may due to the shielding effect of the long alkyl chains over the polyphenylene backbone^[38].

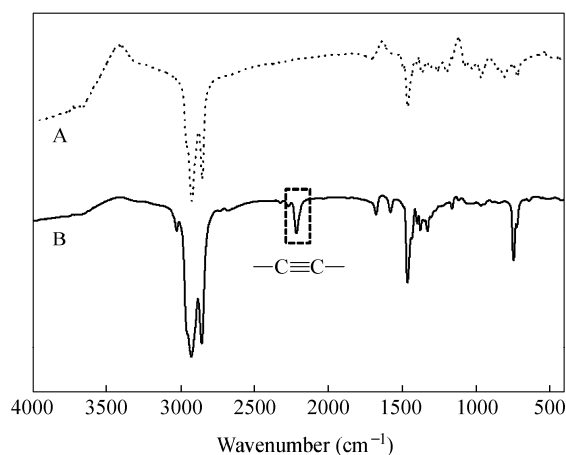


Fig. 2 IR spectra of **1d** (B) and **2d** (A)

Formation of a conjugated backbone was confirmed *via* UV-Vis and PL spectroscopies. As shown in Fig. 4, enediyne precursors **1f** showed an absorbance maxima at around 265 nm and 280 nm, corresponding to the $\pi\text{--}\pi^*$ transition of the conjugated enediyne moiety. After Bergman cyclization, both of the above mentioned features in UV-Vis spectrum disappeared, while a featureless, broad peak showed up. All these polymers showed strong absorptions tailing up to 500 nm, indicating the formation of conjugated systems. The low intensity of the absorption in the long wavelength region is mainly due to the presence of the low molecular weight oligomers

and the difficulty of polyphenylenes to form co-planar conformation due to the steric hindrance of long alkyl chains. In PL spectra, enediyne **1f** barely shows any PL in the visible region. After Bergman cyclization, the polymer **2f** showed blue fluorescence centered at 430 nm, which is clearly related to the formation of conjugated PPP backbone.

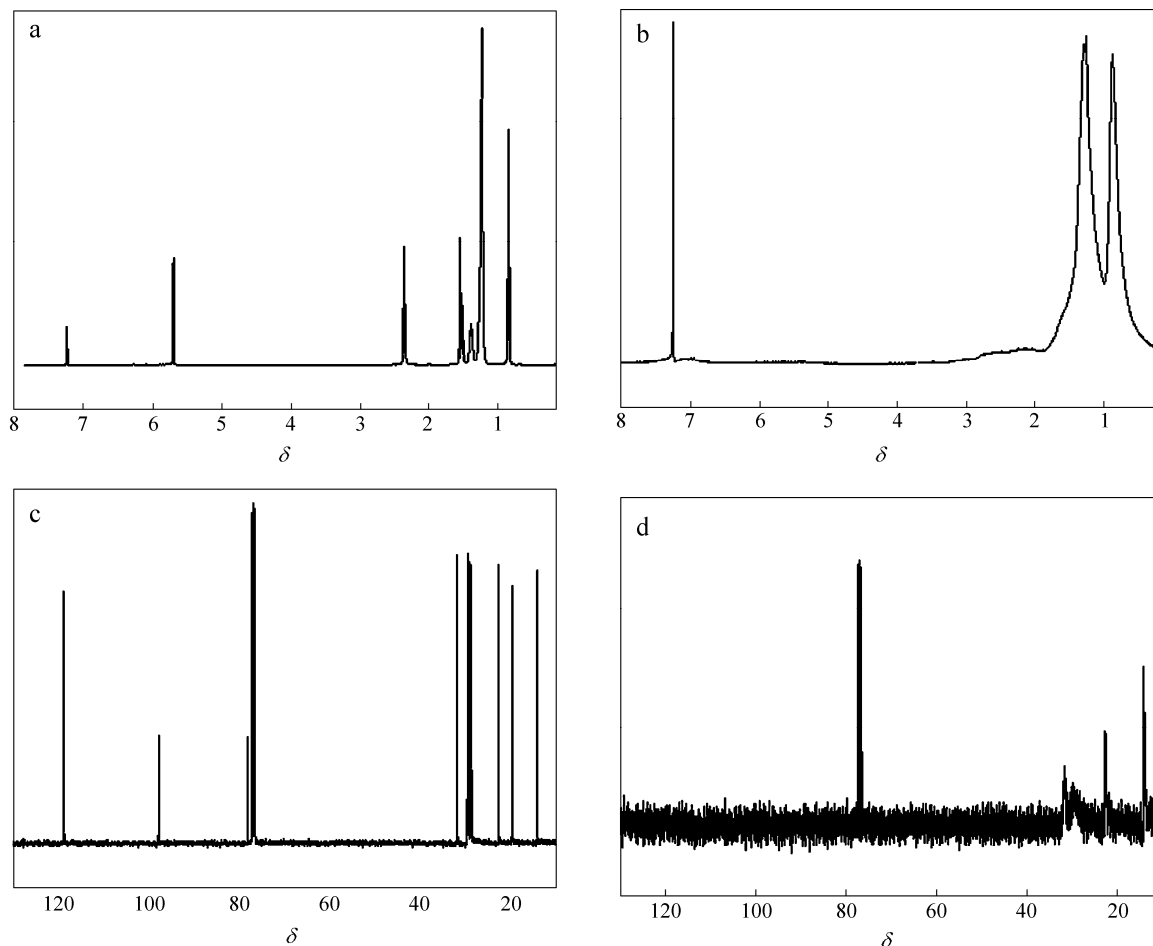


Fig. 3 ^1H - and ^{13}C -NMR spectra of **1d** (a & c) and **2d** (b & d)

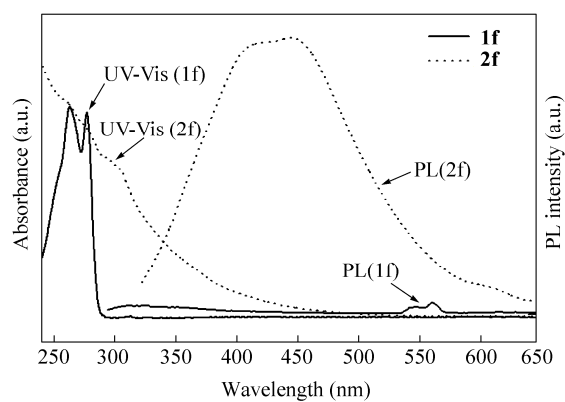


Fig. 4 UV-Vis absorbance and fluorescence emission spectra ($\lambda_{\text{ex}} = 277$ nm) of enediyne precursors **1f** (solid line) and polymer **2f** (dotted line), 10^{-4} mol·L⁻¹ in CH₂Cl₂

The thermal properties of enediynes and polymers were investigated by TGA. As shown in Fig. 5, both the enediyne **1d** and corresponding polymer **2d** possess good thermal stability with the T_{d5} (5% weight loss) and T_{d10} (10% weight loss) around 290 °C and 350 °C respectively in nitrogen atmosphere, revealing their strong resistance to thermolysis at elevated temperature. The weight loss over 300 °C is mainly attributed from the presence of the long alkyl chains. Nevertheless, the good thermal stability of these PPPs is essential for their further applications.

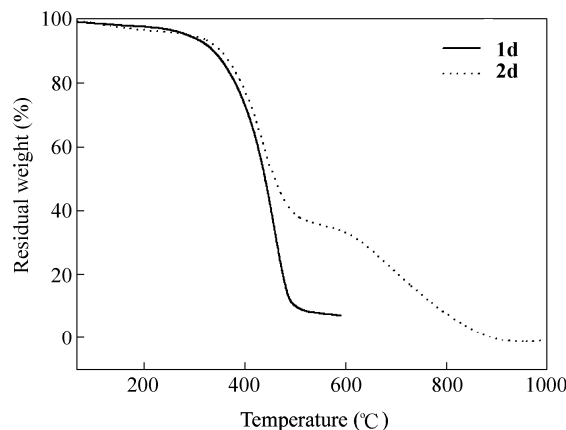


Fig. 5 TGA curves of **1d** (solid line) and **2d** (dotted line)

CONCLUSIONS

In summary, we have successfully synthesized a new series of enediynes with long alkyl chains, which permits us to prepare soluble conjugated PPPs *via* a catalyst-free Bergman cyclization. The occurrence of Bergman cyclization and the formation of PPPs were confirmed with a variety of technique including NMR, GPC, FTIR, and UV-Vis spectroscopy. The PPPs are freely soluble in common organic solvents and show blue fluorescence in solution. They can be further integrated in electronic devices through simple solvent casting process.

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