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Synthesis and characterization of poly(*N*-propargylurea)s with helical conformation, optical activity and fluorescence properties

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ABSTRACT

This article presents novel *N*-propargylurea-based copolymers that contain both carbazole and urea moieties in their side chains. The homopolymer of monomer **1** with a carbazole group exhibited interesting fluorescence properties, while the homopolymer of monomer **2** with a chiral center formed helical structures. The copolymerizations of monomers **1** and **2** provided optically active helical copolymers, which had fluorescence properties, even for the copolymer that contained as low as 20 mol% of monomer unit **1**. Fluorescent and optically active composite films were further prepared based on the copolymers and with poly(vinyl butyl) as the supporting material. This study contributes to the intriguing research field of optically active helical polymers and their practical applications.

1. Introduction

Urea functional groups are very important in organic chemistry. As a well-known denaturant of proteins, urea compounds are frequently used to investigate the folding and unfolding mechanisms of proteins [1]. Cyclic urea derivatives have drawn increasing interest [2] and possess interesting pharmacological properties [3]. Urea derivatives exhibit chirality [4] and organocatalytic functions [5]. They can be used as soft engineering materials [6], habit modifiers [7], optical sensors for anions [8] and gene carriers [9]. With high reactivity and the ability to form stable hydrogen bonds, urea is also frequently used as a linking molecule in the synthesis of functional polymers [10] or segmented copolymers [11].

Carbazole is a hole-transporting and electroluminescent unit. Therefore, it has many unique properties such as light emission [12] and luminescence properties [13]. Polymers containing carbazole moieties in their side chains can be used as photoconductive, electroluminescent, and/or photorefractive materials [14–17], which have numerous potential technological applications, e.g., in high-density optical data storage, multiple image processing, and phase conjugated mirrors [18–20]. Therefore, simultaneous introduction of urea and carbazole moieties to certain polymer backbones for instance polyacetylenes would provide polymers with some novel and interesting properties.

Substituted polyacetylenes possess various intriguing properties such as semiconductivity, nonlinear optical properties, and high gas permeability. In particular, some polyacetylenes can adopt helical conformations [21]. To date, a variety of substituted polyacetylenes such as poly(phenylacetylene)s [22] and poly(*N*-propargylamide)s [23] have been synthesized. We have synthesized a number of helical substituted polyacetylenes including poly(*N*-propargylamide)s [24], poly(*N*-propargylsulfamide)s [25] and poly(*N*-propargylurea) [26–28], either in organic solvents or in aqueous medium [29,30]. Poly(*N*-propargylurea)-containing suitable pendent groups were also found to adopt helical conformations under appropriate conditions [26–28].

In this study, carbazole groups were introduced onto poly(*N*-propargylurea) side chains for the preparation of highly functional polymers which combine the unique properties of helical polymer backbones with the fluorescence properties from carbazole moieties. Such polymers were prepared via copolymerizations of two monomers: achiral monomer **1**, providing the carbazole moiety, and chiral monomer **2**, providing the helical structure. Fluorescent and optically active composite films were further prepared based on the obtained copolymers and with poly(vinyl butyl) as the supporting material. It should be noted that polyacetylenes containing carbazole moieties in their side chains have been synthesized by Tang [31], Masuda [32], and Tabata [33]. However, polyacetylenes that simultaneously contain carbazole and urea groups and the capacity to form ordered

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helical structures have not yet been reported. Such a class of novel polymers undoubtedly integrates the intriguing helical structures and the desirable properties of urea and carbazole functional groups and would be of great interest, especially for practical applications.

2. Experimental section

2.1. Measurements

Melting point (m.p.) was measured by an X-4 micro-melting point apparatus. ^{1}H nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AV600 spectrometer (Bruker, Germany). Fourier transform spectroscopy (FT-IR) spectra were recorded with a Nicolet NEXUS 670 spectrophotometer (Nicolet, USA). Number-average molecular weights (M_n) and molecular weight distributions (M_w/M_n) of the (co)polymers were determined by Gel permeation chromatography (GPC) (Waters 515-2410 system) (Waters, USA) calibrated by using polystyrenes as standards and THF as the eluent (unless otherwise pointed out). Circular dichroism (CD) and ultraviolet–visible (UV–vis) spectra were recorded on a JASCO J-810 spectropolarimeter (Jasco, Japan). Fluorescence spectra were measured on a Varian Cary Eclip sE spectrophotometer (Varian, USA). The thickness of the film was determined with a thickness gauge (Q/DBEF-2-91) (Shanghai Liuling Instrument Plant, China).

2.2. Materials

Propargylamine, bis(trichloromethyl)-carbonate (BTC), poly(vinyl butyral) (PVB) and carbazole were purchased from Aldrich and used as received without further purification. (nbd)Rh$^+$B$^-$(C_6H_5)$_4$ was prepared as reported elsewhere [34]. Solvents were distilled under reduced pressure [22–28].

2.3. Monomer synthesis

The typical monomer synthesis procedure is as follows. To synthesize monomer **1**, carbazole (2 g, 12 mmol) was dissolved in THF (50 ml), and the solution was added dropwise to a solution of BTC (2 g, 6 mmol) in ethyl acetate (150 ml) while stirring at 0 °C for approximately 2 h. Afterwards, the mixture was further stirred at room temperature for another 30 min and then refluxed at 80 °C for about 3 h. Then, ethyl acetate and THF were removed under reduced pressure, producing the corresponding carbamoyl chloride, which was directly used for the next reaction. The carbamoyl chloride was dissolved in ethyl ether (150 ml), into which triethyl amine (1.3 ml, 18 mmol) and propargylamine (0.5 ml, 12 mmol) were subsequently added at room temperature, and the mixture was stirred at room temperature for about 2 h. The solution was washed three times with 2 N HCl and then neutralized by washing with saturated $NaHCO_3$. The resulting solution was dried over anhydrous $MgSO_4$, filtered, and concentrated, producing the target monomer. The crude monomer was further purified by recrystallization with THF and hexane. The data for monomer **1** were as follows: yield-40%, colorless crystal, m.p. 256–258 °C; IR (KBr): 3419 (H–N), 2163 (HC≡), 1697 (C=O), 3048, 1598, 1495, 1447, 1330, 1240, 1138, 997, 925, 853, 725, and 572 cm^{-1}. ^{1}H NMR (DMSO-*d6*, 600 MHz, 25 °C): δ 2.25 (1H, *HC*≡), 3.33 (2H, C≡C*CH$_2$*), 7.14–7.16 (4H, Ar), 7.37–7.39 (2H, Ar), 7.48–7.49 (2H, Ar), and 8.10–8.11 (1H, N–*H*). Calculation for ($C_{16}H_{12}N_2O$): C, 77.42; H, 4.84; N, 11.29. The real values were: C, 77.53; H, 4.79; N, 11.21. Monomer **2** was synthesized according to our previous study [27].

2.4. Polymerization and copolymerization

(Co)Polymerizations were carried out in a glass tube equipped with a three-way stopcock that contained a dry solvent under nitrogen atmosphere. A catalyst (nbd)Rh$^+$B$^-$(C_6H_5)$_4$ (5 mM) was added to a solution of monomers (0.5 M) in THF (2 ml) under nitrogen atmosphere, and the resulting solution was kept at 30 °C for 2 h. After (co)polymerization, the solution was poured into a large amount of hexane to precipitate the polymer out. The solid product was filtered and then dried under reduced pressure. Spectroscopic data of poly(**1**) were as follows: IR (KBr): 3420 (H–N), 2337 (H–C=), 1697 (C=O), 1600, 1447, 1326, 1238, 725, and 564 cm^{-1}. ^{1}H NMR (DMSO-*d6*, 600 MHz, 25 °C): δ 3.31 (2H, C=C*CH$_2$*), 6.45 (1H, *H*–C=), 7.14–7.16 (4H, Ar), 7.36–7.39 (2H, Ar), 7.47–7.48 (2H, Ar), and 8.10–8.11 (1H, N–*H*).

The Spectroscopic data of copolymers were as follows (taking Poly(**1**$_{0.2}$-*co*-**2**$_{0.8}$) as an example): IR (KBr): 3385 (H–N), 2341 (H–C=), 1645 (C=O), and 1523.

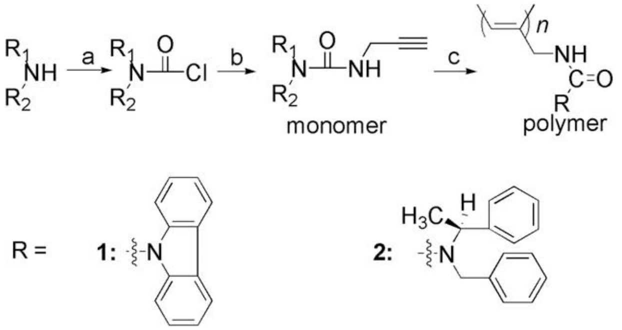
2.5. Preparation of composite films

The copolymers synthesized above were applied to prepare composite films with PVB as supporting material. Copolymers (0.05 g) were dissolved in 20 ml PVB (1 g) $CHCl_3$ solution and cast onto a glass dish. The $CHCl_3$ was then evaporated under ambient conditions and films with a uniform thickness of 55 ± 5 μm were obtained.

3. Results and discussion

3.1. Synthesis and polymerization of N-propargylurea monomers

Monomers **1** and **2** were synthesized according to the strategy outlined in Scheme 1. The major synthetic procedure was as follows. A secondary amine was first reacted with bis(trichloromethyl)-carbonate (BTC) to form carbamoyl chloride, which was then directly reacted with propargylamine to yield the target monomer. The details for the synthesis and purification procedure and the spectroscopic data of monomer **1** are described in the experimental section. Monomers **1** and **2** were polymerized with (nbd)Rh$^+$B$^-$(C_6H_5)$_4$ as the catalyst in THF at 30 °C for 3 h. The relevant details are also presented in the experimental section. Polymerization results for monomer **1** are summarized in Table 1. The results of monomer **2** and poly(**2**) were introduced in our previous article [27]. Poly(**1**) was obtained as an 86% yield and its number-average molecular weight (M_n) was 3700. It was assumed that the low molecular weight resulted from the low coordination ability of the ethynyl group of monomer **1** to Rh catalyst, due to the low electron density [35], and thus the polymerizability of mono-



Scheme 1. Monomer synthesis and polymerization. Reagents: (a) bis(trichloromethyl)carbonate (BTC), ethyl acetate and THF; (b) propargylamine, ethyl acetate; (c) (nbd)Rh$^+$B$^-$(C_6H_5)$_4$ (nbd, norbornadiene), THF.

Table 1
(Co)Polymerization results of monomers **1** and **2**.[a]

Entry	Monomer (**1**/**2**) (mol/mol)	Yield (%)[b]	M_n[c]	M_w/M_n[c]	$[\alpha]_D$[d] (°)
Poly(**1**)	100/0	86	3700	1.36	0
Poly($\mathbf{1}_{0.95}$-*co*-$\mathbf{2}_{0.05}$)	95/5	92	3200	2.11	611
Poly($\mathbf{1}_{0.90}$-*co*-$\mathbf{2}_{0.10}$)	90/10	99	2800	1.82	646
Poly($\mathbf{1}_{0.80}$-*co*-$\mathbf{2}_{0.20}$)	80/20	91	2400	1.21	982
Poly($\mathbf{1}_{0.60}$-*co*-$\mathbf{2}_{0.40}$)	60/40	97	6400	1.94	1107
Poly($\mathbf{1}_{0.40}$-*co*-$\mathbf{2}_{0.60}$)	40/60	90	6700	2.01	1230
Poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$)	20/80	92	8900	2.07	1341
Poly(**2**)	0/100	100	12,700	1.59	1398

[a] With (nbd)$Rh^+B^-(C_6H_5)_4$ catalyst in THF, $[M]_0$ = 0.5 M; $[M]_0/[Rh]$ = 100, 30 °C, 3 h.
[b] Hexane-insoluble part.
[c] Measured by GPC (polystyrenes as standards; THF as eluent).
[d] Measured by polarimeter at room temperature, c = 0.080–0.1 g/dL in $CHCl_3$.

mer **1** was low. The cis content of poly(**1**) was 98%, according to ^1H NMR measurement [24–28], i.e., by the integration ratio between the vinyl proton of the polymer main chain and the other signals. The high cis content was consistent with the preceding studies [24–28], indicating that the Rh catalyst was efficient for synthesizing the polymers with high cis content.

The solubility of poly(**1**) was examined. It was completely soluble in commonly used organic solvents, such as $CHCl_3$, CH_2Cl_2, THF, toluene, DMF, DMSO, and methanol.

Fig. 1 shows the UV–vis spectra of poly(**1**) and monomer **1** measured in $CHCl_3$. Poly(**1**) and monomer **1** show little difference in UV–vis absorption at the wavelength of 290–350 nm. This result suggests that the polyacetylene backbones are not well conjugated, possibly because of the irregular bending and crumpling of the polymer chains [36].

3.2. Preparation and properties of poly(**1**-*co*-**2**)s

(Co)polymerizations of monomers **1** and **2** were carried out for the preparation of helical polymer backbones with carbazole groups in their side chains. The (co)polymerization results are listed in Table 1. The yields of all the copolymers are 90% and above. The molecular weights of the copolymers show a general tendency, i.e., increasing from approximately 3000 to approximately 13,000 with the content of monomer **2** unit. The molecular weights and yields of the copolymers were both higher than those of poly(**1**). In addition, the compositions of the copolymers were in accordance with the monomer feed ratios. Taking poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) as an example, according to the ^1H NMR measurement and the integration ratio between the amine and the methyl protons in the copolymer side chains (in the units of monomer **2**), the proportion of monomer **2** units in the copolymer was found to be 76%. Furthermore, the copolymers were considered to have random structures, supported by our earlier studies [24–28] and the similar investigations from other research groups [37–39]. For example, in the work of Masuda, copolymers of *N*-propargylamides were considered adopting random structures. CD and UV–vis spectra of the poly(**1**-*co*-**2**)s measured in $CHCl_3$ at room temperature are illustrated in Fig. 2.

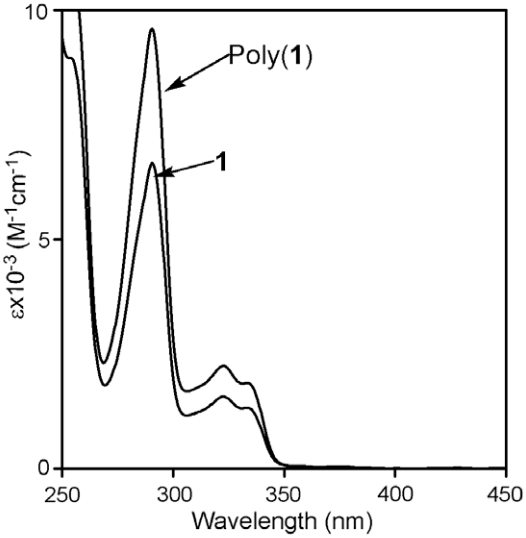


Fig. 1. UV–vis spectra of monomer **1** and poly(**1**) measured in $CHCl_3$ (c = 0.1 mM) at 20 °C.

In Fig. 2a, the copolymers produced UV–vis absorption peaks at 290 nm, 320 nm, and 332 nm, due to the carbazole groups. In addition, UV–vis absorption peaks at 360 nm originated in the polyacetylene main chains. By increasing the content of monomer **1** unit in the copolymers, the intensity of UV–vis absorption peaks at 360 nm decreased. Fig. 2b presents CD spectra of the copolymers. The CD signals of the copolymers at 360 nm demonstrate that the copolymers have large optical activities. Referring to our preceding investigations on poly(*N*-propargylamide)s [24], poly(*N*-propargylsulfamide)s [25] and poly(*N*-propargylurea)s [26–28], we concluded that the copolymers exhibited helical conformations under the examined conditions. Moreover, the CD signals weakened gradually as the content of monomer unit **1** in the copolymers increased, as observed in Fig. 2b. This further indicated that the helical content in the copolymers increased with the content of monomer **2**. Fig. 2b also demonstrates that the formation of helical structures conformed to the widely-known "Sergeants and Soldiers rule" [40]. The specific rotations of copolymers in Table 1 showed the same trend as the CD signals, which also demonstrated that the copolymers possessed very high optical activities.

Fig. 3 shows Kuhn's dissymmetry ratios [41] ($g_{abs} = \Delta\varepsilon/\varepsilon$, in which $\Delta\varepsilon = [\theta]/3298$) in a series of (co)polymers at approximate 360 nm, as a function of monomer **2** unit composition, in order to quantitatively evaluate the chiral molar composition response of the Cotton effects. It appears that only 5 mol% of monomer **2** unit in the copolymers completely determines the overall screw sense, as shown in Fig. 2. With the composition of monomer **2** unit increasing, the *g* value rises, indicating that the preferences of helical screw sense of the copolymer were increased. When the composition of monomer **2** unit is more than 10 mol%, the *g* value was almost constant. It can be concluded that the copolymers maintain their preferential helical screw sense in the region of monomer **2** units above 10 mol%.

Fig. 4 shows the fluorescence spectra of monomer **1** and poly(**1**). Monomer **1** and poly(**1**) exhibit fluorescence at 360 nm upon excitation at 332 and 324 nm, respectively. The fluorescence properties

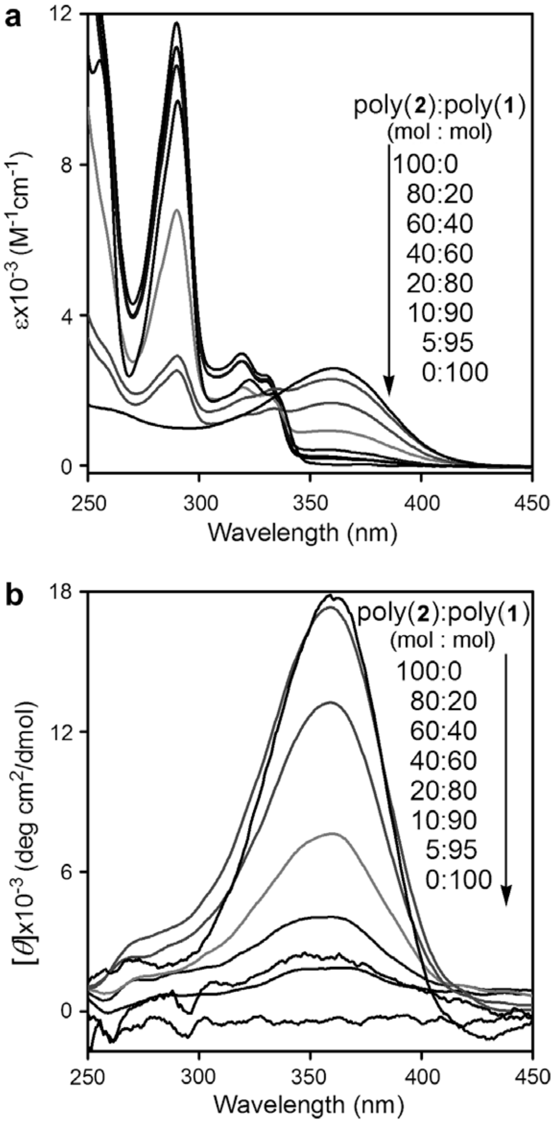


Fig. 2. UV–vis (a) and CD (b) spectra of poly(**1**-*co*-**2**)s measured in $CHCl_3$ ($c = 0.1$ mM) at 20 °C.

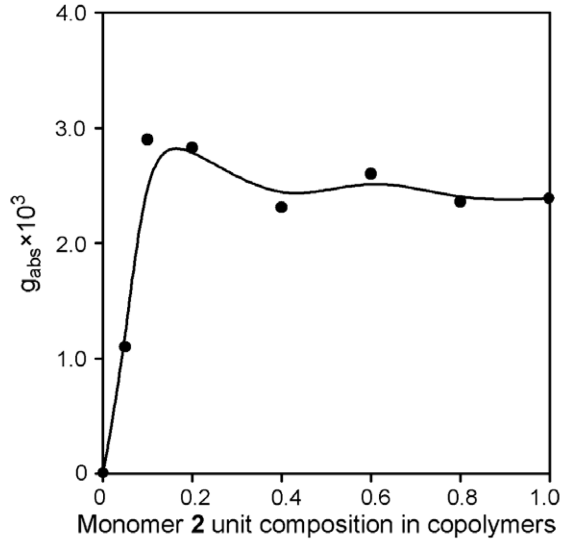


Fig. 3. Plots of the *g* values of (co)polymers versus monomer **2** unit composition (calculated from data of Fig. 2).

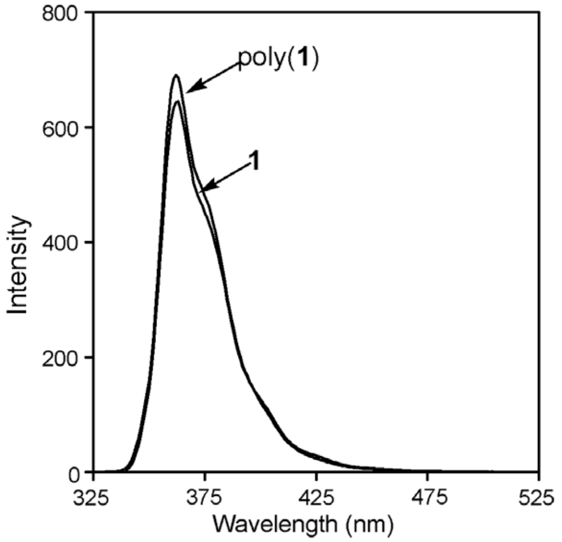


Fig. 4. Fluorescence spectra of monomer **1** and poly(**1**) measured in THF, excited at 332 and 324 nm, respectively.

of monomer **1** and poly(**1**) were due to the carbazole groups in their side chains.

Having helical (co)polymers with carbazole groups in their side chains available, we further characterized the (co)polymers by fluorescence spectroscopy. Copolymers have fluorescence properties, even for the copolymer containing monomer unit **1** as low as 20 mol% [poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$)], as presented in Fig. 5. Poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) exhibits fluorescence at 430 nm upon excitation at 390 nm. A red shift can be observed in poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) (Fig. 5) when compared to poly(**1**) (Fig. 4). Three are some factors leading to the shift, for instance the intramolecular hydrogen bonds and the synergic effects between the pendent groups especially in the copolymers. Accordingly, poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) not only adopted a helical conformation, but also exhibited fluorescence properties.

3.3. Optical activity and fluorescence properties of composite films

To attest to the practical uses of the fluorescent poly(*N*-propargylurea)s with chiroptical property, composite films consisting of copolymers and PVB were prepared. PVB acts as a supporting material for the optically active helical polymers. The composite film from poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) (as a representative) and the pure PVB film were characterized with both UV–vis and CD spectroscopies, as presented in Fig. 6. Compared to the pure PVB film with minor UV–vis absorption and no CD signal at 250–450 nm, the composite film showed substantial UV–vis absorption peaks at 290, 320, and 332 nm, originating from the carbazole groups. The composite film also showed a peak at 375 nm that was the copolymer's main chains. The CD spectra showed a bi-signal Cotton effect and similar shapes, as presented in Fig. 6b. The CD signal at around 325 nm and 375 nm strongly demonstrated that the composite film possessed very high optical activity. The bi-signal Cotton effect assumed the formation of helical supramolecular structure in the composite films of the copolymers, similar to polymer aggregates [42].

The prepared composite films were also subjected to fluorescence spectroscopy measurements, and the result is illustrated in Fig. 7. The composite film exhibited fluorescence at 420 nm upon

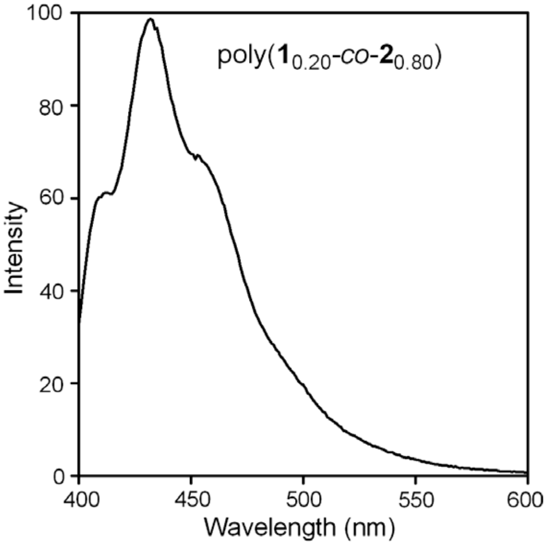


Fig. 5. Fluorescence spectrum of poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) measured in THF, excited at 390 nm.

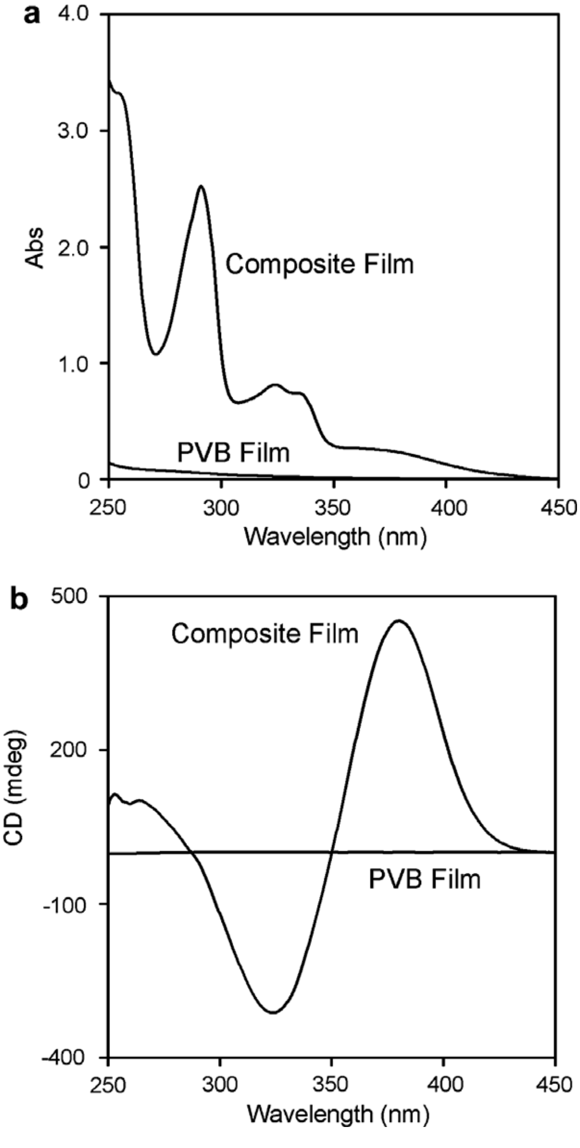


Fig. 6. UV–vis (a) and CD (b) spectra of the composite film containing poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$) and the pure PVB film.

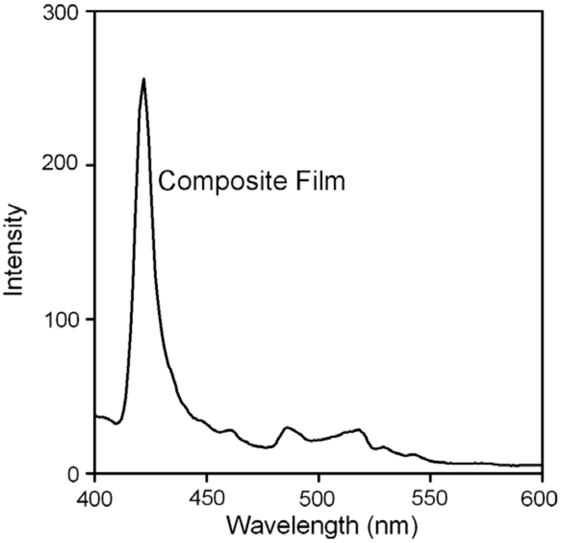


Fig. 7. Fluorescence spectrum of composite film containing poly($\mathbf{1}_{0.20}$-*co*-$\mathbf{2}_{0.80}$), excited at 370 nm.

excitation at 370 nm; this wavelength was similar to that of the corresponding copolymer. The results of CD and fluorescence spectroscopy measurements demonstrated that the composite films exhibited both optical activity and fluorescence property. The novel (co)polymers and composite films reported here are thus considered having potential applications for instance as optically active luminescent materials emitting circular polarized light. The relevant investigations are in progress.

4. Conclusions

A poly(*N*-propargylurea) compound with carbazole groups (poly(**1**)) in its side chains was successfully prepared by polymerizing the monomer in the presence of (nbd)$Rh^{+}B^{-}(C_6H_5)_4$ as the catalyst. Poly(**1**) had fluorescence properties. The copolymers of monomers **1** and a chiral monomer **2** showed strong CD signals, indicating that the copolymers had large optical activities and adopted helical conformations with a predominantly one-handed screw sense. The copolymers had considerable fluorescence properties. Composite films consisting of the copolymers and PVB exhibited optical activities and fluorescence properties. These investigations allow further design and development of multifunctional polymers and facilitate the use of substituted polyacetylenes in practical applications.

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