Molecular structure modulated properties of azobenzene-substituted polydiacetylene LB films: Chirality formation and thermal stability

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ABSTRACT

Langmuir–Blodgett (LB) films of three novel azobenzene-substituted diacetylene monomers (DA1, DA2 and DA3) were fabricated and their optical and chiroptical properties were investigated in detail by ultraviolet–visible (UV–vis) spectra and circular dichroism (CD) spectra. Achiral DA1 molecules could form chiral LB films through overcrowded packing of the azobenzene moiety, while achiral DA2 and DA3 molecules not. When exposed to left- or right-handed circular polarized UV light (CPUL), striking left- or right-handed (opposite) CD signals for azobenzene chromophores and polydiacetylene chains were observed for the polymerized DA1 (PDA1) and DA2 (PDA2) LB films. However, DA3 LB films could hardly be polymerized in this case, and only striking opposite CD signals for azobenzene chromophores could be observed. It was demonstrated that the intermolecular steric hindrance and irregular arrangement of azobenzene chromophores were not favorable for the topo-polymerization and chirality formation of polydiacetylenes backbone. Further, the effects of thermal treatment on the supramolecular chirality of above three LB films were studied. Strong collective noncovalent interactions (π–π stacking) were believed to be responsible for the thermal stability of chiral supramolecular assemblies.

1. Introduction

The UV-induced polymerization of multifunctional monomers has been widely applied in various industrial fields mainly in the production of films, inks, and coatings [1–6]. Polydiacetylene (PDA) is one of the most promising topochemical polymerization materials, which have many potential applications in biosensors [7], pathogenic agents [8] and optical [9], electrical devices [10,11]. Moreover, PDA is well-known as a quasi one-dimensional (1D) conjugated polymer, specific carrier transport is expected due to the 1D electronic state. These interesting electronic behaviors attract us to the intensive researches. In any case, structural control of the PDA backbone must be an essential process to bring out the useful function from PDA effectively. In chiral system, symmetric mirror plane disappears, and interaction between magnetic field and chiral materials plays an important role due to the pseudo-vector nature of magnetic field. Thus, introduction of chirality into the conjugated materials such as polydiacetylene is expectative to fruition of novel organic devices with special electro-magnetic functions. Liu's group and we have reported the successful

preparation of chiral structure in polydiacetylene monolayer and multilayer [12–14], where the presence of metal ions in subphase was a key to the chiral formation and chiral structure could only be accidentally obtained. In our previous work [15,16], we also reported the successful enantioselective polymerization of poly-diacetylene using circular polarized UV light (CPUL) where photo-sensitive nature of polydiacetylene and chiral nature of CPUL were effectively used. And the photo-reversible switching between opposite chirality of polydiacetylene backbone has been realized [17,18]. However, the detailed mechanism of the chiral introduction into the polydiacetylene backbone is still not very clear; especially the molecular structures effect on chiral formation and thermal stability was not extensively studied.

Investigations on the self-organization of amphiphiles into different supramolecular aggregates structures have attracted much attention [19–25]. Theoretical studies pioneered by Israel-achvili et al. and Hyde have demonstrated that controlling the shape of individual amphiphilic molecules by tuning the size of the hydrophobic part relative to the hydrophilic part allows variation of the topology of the self-assembled structures [26–28]. In this work, three novel diacetylene monomers were synthesized and the effect of molecular structure on the polymerization behavior, chiral formation and thermal stability were investigated in detail.

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The azobenzene mesogen is a well-known photosensitive chromophore, which undergoes photo-induced *trans* to *cis* (or *cis* to *trans*) isomerization under suited light irradiation [29]. A series of chiral molecular assemblies and/or chiral polymers from azobenzene derivatives have been fabricated with azobenzene units bearing intrinsic chirality and several chiroptical devices have been proposed [30–34]. There are also interesting reports that achiral azobenzene derivatives could be photo-induced into chiral assemblies by circularly and/or elliptically polarized beam irradiation and the photo-induced configuration control of the azobenzene derivative could be fabricated the optical active supramolecular assemblies from achiral azobenzene derivative [35–39]. In this paper, three accessible azobenzene mesogen-substituted diacetylene monomers (DA1, DA2 and DA3) LB films were fabricated and the links among molecular structure, the organization and chiral properties of the resulting polydiacetylene assemblies LB films were established. Molecular structure and intermolecular interaction were expected to modulate the stacking structure at both the molecular and the supramolecular levels, leading to different polymerization behavior, chirality and thermal stability for the resulting LB films. Furthermore, the effect of photoisomerization of the azobenzene chromophore on the chirality formation of the resulting LB films were investigated in detail. This work not only provides rational design of novel chiral PDA assemblies, but also is of great fundamental value for the understanding of the chiral formation mechanism of PDA derivative materials.

2. Experimental sections

2.1. Sample preparation

DA1, DA2 and DA3 were synthesized in analogy to the previous procedure [40,41] and their molecular structures were shown in Fig. 1. The DA1, DA2 and DA3 were dissolved in AR grade chloroform with the concentration of 1 mg/mL. Subphase was pure water (18.2 MΩ). All LB deposition experiments were carried out at 20 °C (±0.5 °C) on a computer-controlled Langmuir film balance (600 mm × 150 mm in length and width, 10 mm in depth). The substrate for LB deposition was quartz glass. The number of deposited layers was 100. A 16 W UV lamp (λ = 254 nm) hung 10 cm above the LB films was used for UV-polymerization. And the circularly polarized UV light (CPUL) (313 nm) generated using Babinet–Soleil prism from ultra-high pressure mercury lamp was used for enantioselective polymerization [15–17].

2.2. Characterization

The ultraviolet–visible (UV–vis) spectra of samples on quartz substrate were measured using a spectrometer (Shimadzu: UV 2100). CD spectrum was measured using JASCO CD spectrometer J-715. The possible effect of linear dichroism of the film was removed by rotating the film plate around the incident light [42,43].

3. Results and discussion

3.1. Preparation of three azobenzene-substituted diacetylene monomer LB films

In order to investigate the above-mentioned issues, three azobenzene-substituted diacetylene monomers (DA1, DA2 and DA3) monolayers formed on a water surface could be transferred onto solid substrates at 22 mN/m by a horizontal lifting method under darkness. Fig. 2 showed UV–vis spectra of DA1, DA2 and DA3 both of the isomers in chloroform solutions and the LB films. The UV–vis spectrum of *trans*-form DA1, DA2 and DA3 exhibited strong absorptions peaks at 370 nm, 453 nm and 453 nm, respectively, which were originated from the $\pi-\pi^*$ transition the azobenzene chromophore. The absorption of their 100-layer LB films showed broadened band centered around 360 nm, 401 nm and 403 nm, respectively, which were blue-shifted with respect to that of the $\pi-\pi^*$ transition in chloroform solution. These excitonic absorption shifts could be understood as an indication that DA1, DA2 and DA3 formed H aggregates by face-to-face stacking of azobenzene functional groups under the given experimental conditions. When DA1, DA2 and DA3 LB films were subjected to CD measurements, it was interesting to note that DA1 LB films showed an obvious Cotton effect although the monomer DA1 itself was achiral. In other words, the macroscopic chirality of DA1 LB films was introduced as a result of a cooperative stereo regular arrangement of azobenzene functional groups. In the present case, the $\pi-\pi$ stacking between adjacent DA1 molecules could also contribute to the spontaneous overcrowded packing of the functional groups into a helical sense, and thus the transferred LB films showed a chiral-optical effect. It should be noted here that although we could always get the CD signals, DA1 samples with the same UV–vis absorbance might show different CD intensity or even opposite CD signals by chance in different batches. As we know, the chance of the right or left-handed helical structure forming of the backbone should be equal, and under normal case no chiral assemblies could be obtained. The
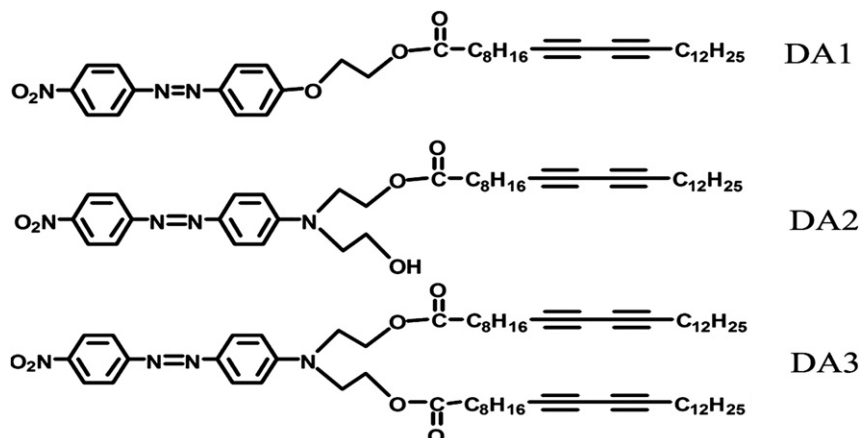

Fig. 1. Illustration of molecular structure for the three azobenzene-substituted diacetylene monomers (DA1, DA2 and DA3).
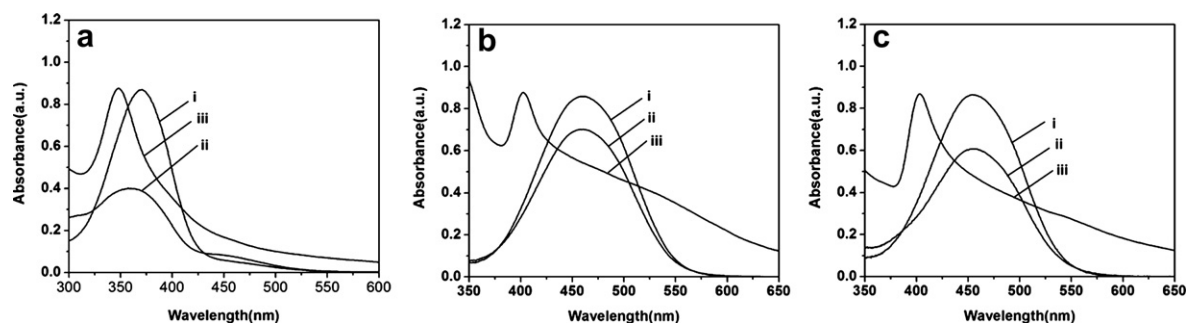
Fig. 2. UV/vis spectra of monomer (a) DA1, (b) DA2 and (c) DA3 (i: *trans*-form in chloroform solution, ii: *cis*-form in chloroform solution, iii: LB films).

symmetry could be broken at the point where the monomer molecules were compressed to an overcrowded array, and one of the enantiomers was predominant by chance. As for DA2 and DA3 LB films, short hydroxyethyl or long side chains precluded the very uniform ordered alignment of azobenzene mesogens as those in the films derived from DA1. Namely, due to the intermolecular steric hindrance and the disordered arrangement of DA2 and DA3, no closely cooperative arrangement could be formed. Therefore, we could not observe the supramolecular chirality for DA2 and DA3 LB films (as shown in Fig. 3).

3.2. Enantioselective polymerization of the three monomer LB films

Above three LB films were exposed to normal UV light irradiation for 8 min after deposition. As shown in Fig. 4a, the LB films derived from DA1 were substantially polymerized with intense absorption maxima at about 547, 580, 625 nm. While the LB films derived from DA2 were polymerized into red phase directly with absorption maxima at about 487 nm and 520 nm (as shown in Fig. 4b). DA3 LB films could hardly be polymerized and no new absorption peaks were observed after irradiation (as shown in Fig. 4c), which could be ascribed to the extraordinary overcrowded packing state of the azobenzene chromophores. And in this case, it did not meet the strict topochemical requirements for polymerization [44] (the monomer diyne units should be arranged with a repeat distance (r) of ∼5 Å and an orientation angle (θ) of ∼45° relative to the translation axis). After UV irradiation (see Figure S1 in supporting information), the resulting polymerized films derived from DA1 and DA2 showed characteristic Raman vibrational bands at 2115, 2111 and 1512, 1508 cm^{-1}, respectively, corresponding to C≡C and C=C stretching vibrations of the polymer backbone. It was demonstrated that the DA1, DA2 monomers all have been successfully polymerized into usual ene-yne structure via 1, 4-addition polymerization reactions after UV irradiation. Then, the resulting LB films after UV irradiation were subjected to the CD characterization. No Cotton effect for the polydiacetylenes chains

could be observed for the resulting polymerized DA1 and DA2 LB films (Fig. 4a and b). All above results indicated that helical structure of polydiacetylenes backbone could not form under the given experimental conditions.

In our previous work [15–17], we reported the successful enantioselective polymerization of polydiacetylene using CPUL. Herein, above three azobenzene-substituted diacetylene monomers LB films were subjected to CPUL irradiation for 20 min. To avoid the variation between individual deposition batches, monomer LB films for left- and right-handed CPUL irradiation were prepared at the same batch. As expected, CD spectra for the DA1 LB films polymerized by left- and right-handed CPUL clearly showed opposite CD signals not only for azobenzene chromophores, but also for polydiacetylene chains as shown in Fig. 5a. That is, the film polymerized by left-handed CPUL showed negative Cotton effect at 630 nm (for polydiacetylene chains) and positive Cotton effect at 355 nm (for azobenzene chromophores); while the film polymerized by right-handed CPUL showed positive Cotton effect at 630 nm (for polydiacetylene chains) and negative Cotton effect at 350 nm (for azobenzene chromophores). Namely, left- and right-handed CPUL distinguishably produced the PDA1 LB films with opposite chirality. Unlike the normal UV irradiation case, this enantioselective polymerization of DA1 LB films should be a deterministic process and could be completely controlled. Even DA1 LB films from different batches gave opposite CD signals, they would show similar CD signals for both azobenzene chromophores and polydiacetylene chains upon CPUL polymerization (see Figure S2 in the supporting information). Presumably, simultaneous photo-induced rearrangement of azobenzene chromophores in the side chains would form helical structures with the same handedness direction at the initial stage of the polymerization (see Figure S3 in the supporting information). Thus the helical direction of above chiral structure formed in PDA1 LB films would be the same as the handedness of CPUL, no matter what directions of packing structure of azobenzene chromophores in DA1 LB films.
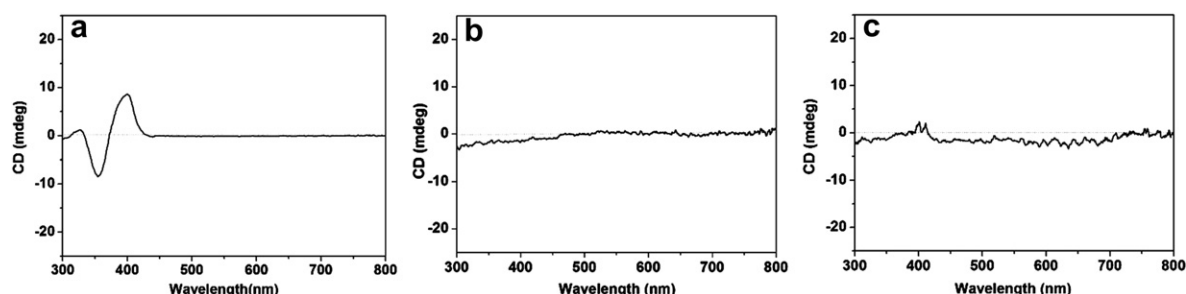

Fig. 3. CD spectra of (a) DA1, (b) DA2 and (c) DA3 LB films before UV irradiation. The number of deposited layers was 100.
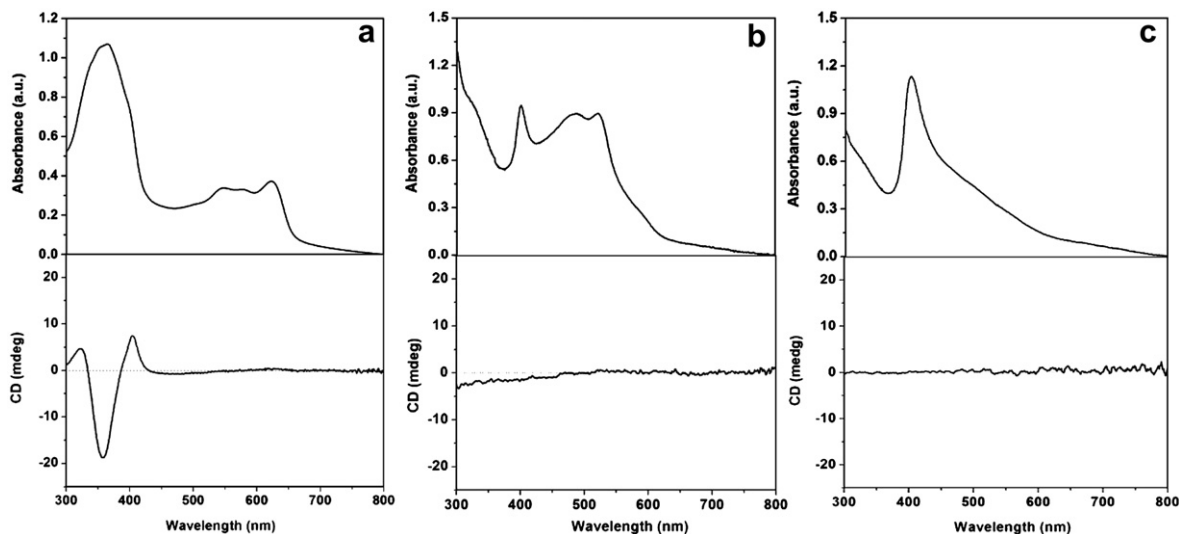
Fig. 4. UV–vis and CD spectra of (a) DA1, (b) DA2 and (c) DA3 LB films after normal UV light irradiation.

After CPUL irradiation, PDA2 LB films (Fig. 5b) also exhibited CD signals for azobenzene chromophores and polydiacetylene chains and showed opposite CD signals when treated with left- and right-handed CPUL. And similar correlation between the CD signals originated from azobenzene chromophores and polydiacetylene chains could be observed: the film with the positive Cotton effect at 367 nm for azobenzene chromophores would show negative Cotton effect at 530 nm for polydiacetylene chains; the film with the negative Cotton effect at 367 nm would show positive Cotton effect at 530 nm. As mentioned above, DA3 LB films could hardly be polymerized under the given condition, and only obvious Cotton effect for the azobenzene chromophores could be observed (as shown in Fig. 5c).

3.3. Effect of photoisomerization of the azobenzene chromophore on the chirality formation

The azobenzene mesogen is a well-known photosensitive chromophore, which undergoes photo-induced *trans* to *cis* (or *cis* to *trans*) isomerization under suited light irradiation. DA1 undergoes photo-induced *trans* to *cis* isomerization under 365 nm light irradiation, DA2 and DA3 undergo photo-induced *trans* to *cis* isomerization under 435 nm light irradiation [45]. It was anticipated that the photoisomerization of the azobenzene chromophore may influence the molecular packing of the assemblies and the chirality formation of the resulting LB films.

In order to investigate the above-mentioned issues, DA1, DA2 and DA3 monomers were dissolved in chloroform solution as a *trans*-form. To obtain *cis*-form monomer in solution, the chloroform solution of *trans*-DA1 was irradiated with 365 nm UV light (435 nm light for *trans*-DA2 and *trans*-DA3) for 10 min to achieve the photoisomerization from *trans*-DA1 to *cis*-DA1. The solutions of *cis*-samples in chloroform were spread on a water surface and the resulting LB films were investigated in detail. When the *cis*-form DA1, DA2 and DA3 LB films were subjected to CD spectral measurements, no CD signals could be observed for the *cis*-form LB films (as shown in Fig. 6a–c). It was demonstrated that the configuration of azobenzene chromophore
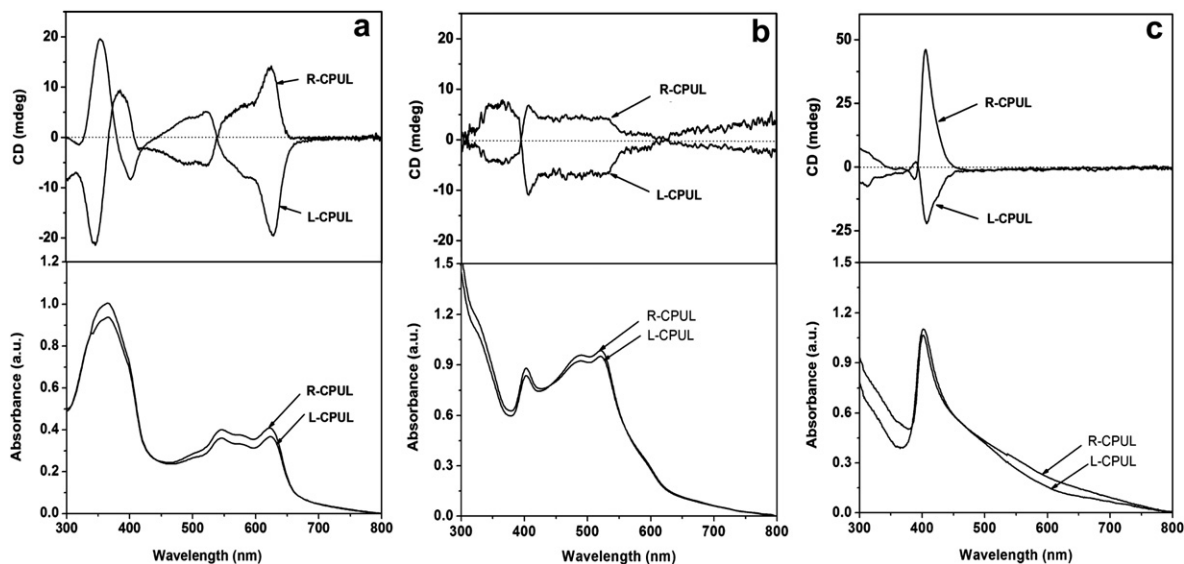

Fig. 5. UV–vis and CD spectra of (a) DA1, (b) DA2 and (c) DA3 LB films after left- and right-handed CPUL irradiation. The number of deposited layers was 100.
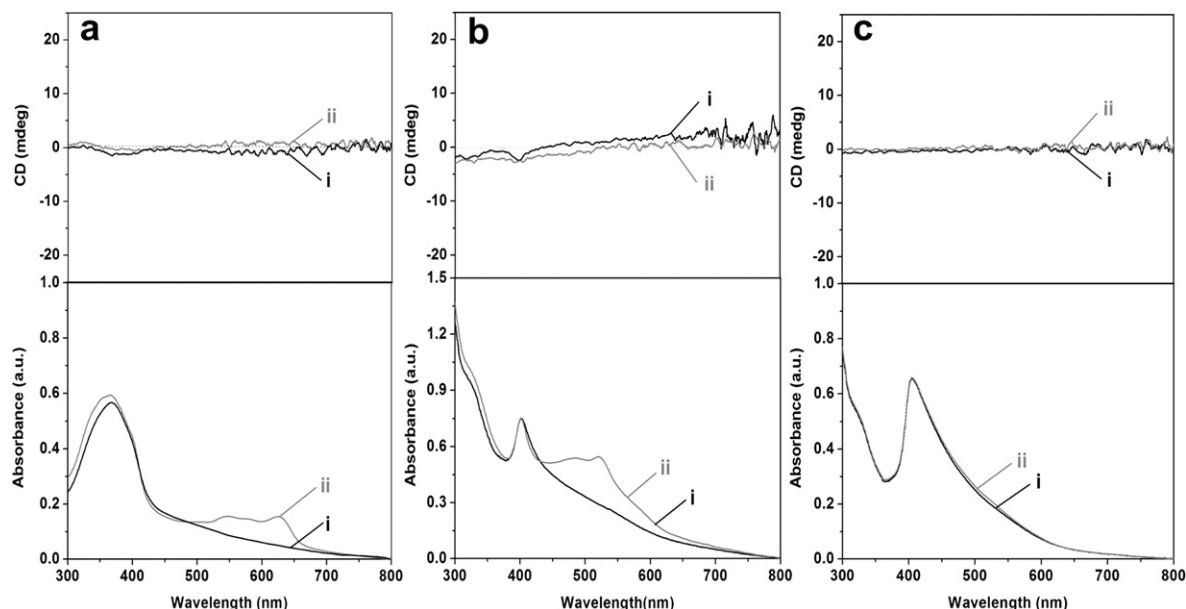
Fig. 6. UV–vis and CD spectra of *cis*-DA1 (a), *cis*-DA2 (b) and *cis*-DA3(c) LB films before and after left-handed CPUL irradiation. The number of deposited layers was 100.

played an important role in the supramolecular chirality formation for the transferred films.

Above three *cis*-form monomers LB films were subjected to Left-handed CPUL irradiation for 20 min. Cis-samples DA1 and DA2 could be polymerized, with characteristic absorption peaks of polydiacetylenes as shown in the UV–vis spectra (Fig. 6a and b). While *cis*-form DA3 LB films could hardly be polymerized as shown in Fig. 6c. It was interesting to note here that no Cotton effect either for azobenzene chromophore or the polydiacetylenes chains could be observed for the resulting PDA1, PDA2 and DA3 LB films (Fig. 6a–c). Based on above data, a possible mechanism could be proposed to explain this phenomenon (as shown in Fig. 7). In fabricating the LB films, the packing structures of these two isomers of the azobenzene unit could be different due to their different spatial configurations. The *trans*-azobenzene unit had a planar molecular configuration, whereas the *cis*-azobenzene was bent-shaped. When these two forms of the molecules were arranged through the air/water interface, the *trans*-azobenzene packed in an H-like aggregate, while the *cis* isomer formed a J-like

aggregate [39,46]. The linear rod-like structure of *trans*-azobenzene favored the cooperative packing in a helical sense due to the strong intermolecular interactions and the steric hindrance of the adjacent molecules. Thus, a certain enantiomer was predominantly formed and the supramolecular assemblies with supramolecular chirality were obtained. In the case of *cis*-form samples, no cooperative arrangements could be gained due to the complicated intermolecular steric hindrance and the disordered loose arrangement of the molecules. Therefore, we could not observe the supramolecular chirality from the *cis*-form monomer LB films and the LB films after CPUL irradiation.

3.4. Thermal stabilities of the chirality of above three azobenzene-substituted diacetylene monomers LB films after CPUL irradiation

The chirality of above three azobenzene-substituted diacetylene monomers LB films after CPUL irradiation were due to the stereo regular arrangement of both azobenzene chromophores in the side chains and polydiacetylene backbone. It is different from molecular chirality, where the chiral center or axis exists within a molecule. Usually, supramolecular chirality is not as stable as molecular intrinsic chirality. To check the thermal stability of above films, we measured the CD spectral changes of above films upon annealing treatment (heating at 120 °C for 10 min and then cooled to 25 °C in N_2 ambience). Interestingly, PDA1 LB films showed obvious CD signals intensity enhancement not only for azobenzene chromophores, but also for polydiacetylene chains (as shown in Fig. 8a). This intensity enhancement of the CD signals of PDA1 LB films should be ascribed to the rearrangement of azobenzene chromophores, which reasonably produced a more ordered chiral structure of the same sign as the starting one [17]. Before thermal treatment, only small parts of azobenzene chromophores in the side chains formed chiral supramolecular arrangement and showed relative weak CD signal. During the annealing process, these small amounts of chiral assemblies of azobenzene chromophores in the side chains could induce other parts of azobenzene chromophores in the side chains to follow their chirality in the same manner through their strong π–π stacking interaction, which subsequently lead to the resulting rearrangement of polydiacetylene chains and more ordered helical structure formation of the polymer backbone as
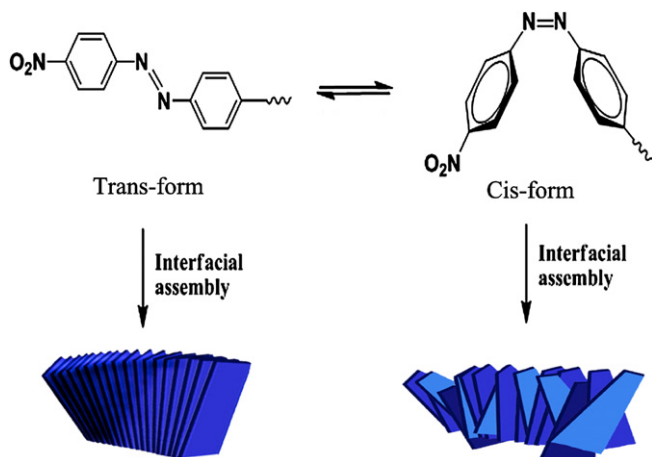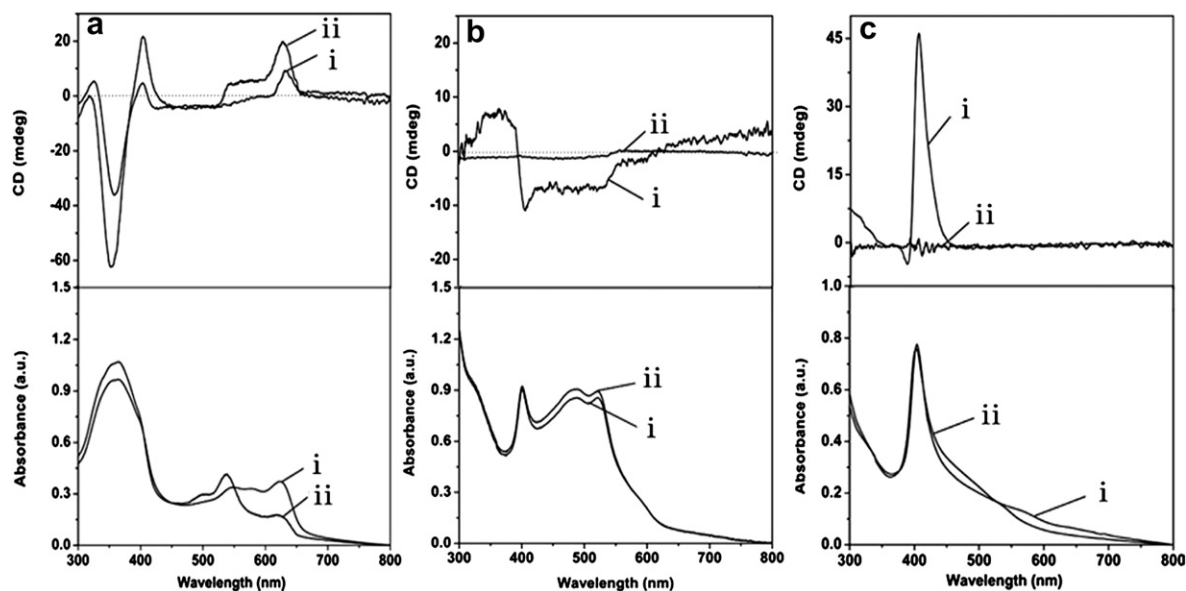

Fig. 7. Proposed model of the formation of optically active and inactive supramolecular assemblies from *trans*-form sample and *cis*-form sample.

Fig. 8. UV—vis and CD spectra of (a) DA1, (b) DA2 and (c) DA3 LB films before and after annealing at 120 °C in N$_2$ ambience for 10 min. The number of deposited layers is 100. (i: before, ii: after).

illustrated in Fig. 9a, what is called "sergeants and soldiers" principle [47]. Obvious peaks shift for the corresponding polydiacetylene chains could be observed for CD and UV absorption spectra measurements of PDA1 films after annealing treatment (as shown in Fig. 8a), which was due to the partial blue-to-red phase

transition of PDA1 during annealing process as reported in our previous work [40].

Unlike PDA1 LB films, LB films derived from DA2 and DA3 showed chirality eraser during annealing treatment (as shown in Fig. 8b and c), which should be ascribed to the relatively weak intermolecular
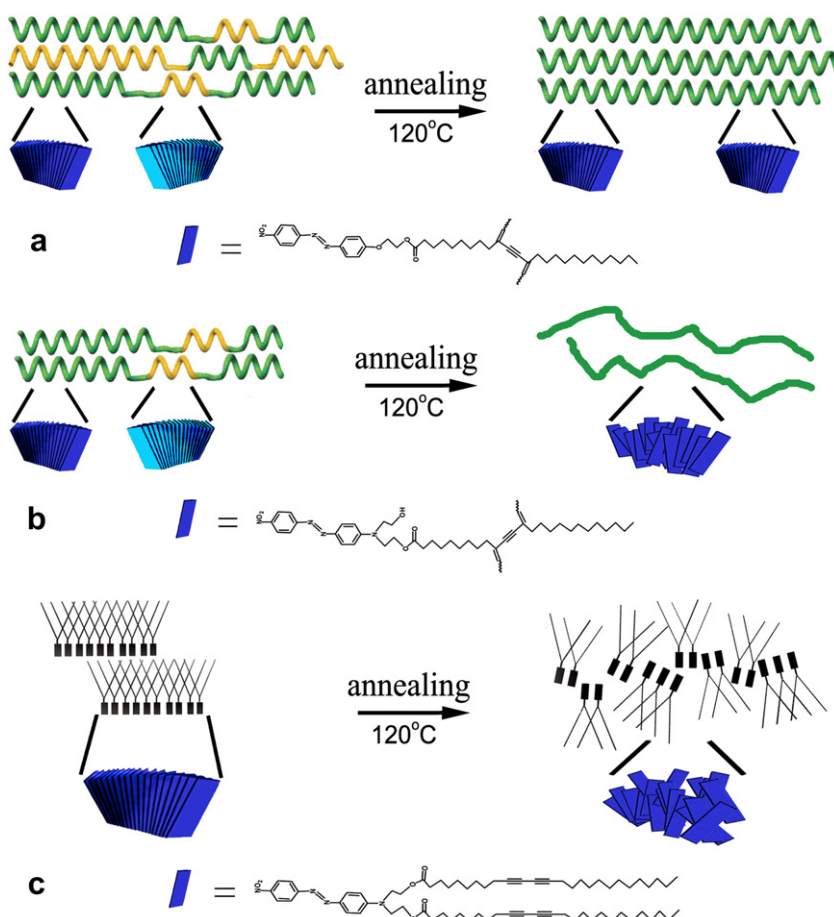
Fig. 9. Proposed models for the LB films derived from (a) DA1, (b) DA2 and (c) DA3 upon annealing at 120 °C for 10 min after CPUL irradiation.

interactions among repeat units. Upon heating, short hydroxyethyl side groups would induce the azobenzene chromophores and the polydiacetylenes into a more disordered arrangement state as illustrated in Fig. 9b. As for DA3 LB films, long side chains and the irregular packing structure offered little intermolecular among side chains. And the originated helical conformation would be easily destroyed into a disordered state upon heating as illustrated in Fig. 9c. All above results indicated that strong intermolecular interactions (e.g. $\pi-\pi$ stacking and covalent interactions) were believed to be responsible for high thermal stability of chirality for PDA1 LB films.

4. Conclusion

In summary, we succeed in synthesis of three novel azobenzene-substituted diacetylene monomers (DA1, DA2 and DA3) and the links among molecular structure, the organization and chiral properties of the resulting polydiacetylene assemblies LB films were established. Achiral DA1 molecules could form chiral LB films through over-crowded packing of the azobenzene moiety, while achiral DA2 and DA3 molecules not. When exposed to left- and right-handed circular polarized UV light (CPUL), obvious opposite CD signals for azobenzene chromophores and polydiacetylene chains were observed respectively of polymerized DA1 (PDA1) and DA2 (PDA2) LB films. However, DA3 LB films could hardly be polymerized in this case, and only striking opposite CD signals for azobenzene chromophores could be observed. The intermolecular steric hindrance and irregular arrangement of the molecules was not favorable for the topo-polymerization and chirality formation of polydiacetylenes. Further, the effect of photoisomerization of the azobenzene chromophore on the chirality formation of the resulting LB films was investigated in detail. Upon annealing to 120 °C, the intensity of the Cotton effect for PDA1 LB films increased greatly, while chirality erasure was observed for PDA2 and the resulting DA3 films. Strong collective noncovalent interactions ($\pi-\pi$ stacking) were believed to be responsible for the thermal stability of chiral supramolecular assemblies.

Acknowledgement

Appendix. Supplementary data

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.polymer.2010.03.029.

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