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Synthesis and Fluorescent Behaviors of Polymers Bearing Stilbene Derivatives

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A series of novel acrylate monomers bearing stilbene segments with D-π-A structure, 4-methacrylate-4′-nitro stilbene (NS), 4-(6-methacrylate)hexyl-4′-nitro stilbene (HNS) and 4-(6-methacrylate)hexyl-2,3-dimethoxy-4'-nitro stilbene (DMHNS), were synthesized by the Witting reaction and polymerized by free radical polymerization. These stilbene-containing monomers and polymers were fully characterized, and their fluorescent properties in solution and solid state were also investigated. The emission band of monomers and corresponding polymers in solution also showed a remarkable effect with increasing polarity of the solvent. More importantly, the fluorescence emission could be adjusted from green to orange and even red regions by forming donor-π-acceptor (D-π-A) construction.

Keywords: Functional monomer, stilbene, D-π-A structure, fluorescence tuning, polymerization

1 Introduction

Stilbene derivatives have attracted great attention due to their significant potential applications, such as fluorescent brighteners (1, 2), chemical sensors (3, 4), organic light emitting diodes (OLED) (5–7), etc. Most of these compounds show blue or green emissions. However, towards the goal of full-color display, it is worthwhile to synthesize the kinds of stilbene derivatives with orange and red fluorescence (8, 9).

In addition, compounds with a donor-π-acceptor (D-π-A) structure have attracted great attentionsbecause of their outstanding optical properties (10, 11). The fluorescent property of stilbene derivatives may show some interesting emission tuning by the formation of D-π-A structure. Guoqiang Chen and coworkers (12) reported a series of D-π-A type stilbene dyes and found that the fluorescence emission would be blue-shifted when the electron-donating effect of the substituents in dye molecules was decreased. Fuyou Li and coworkers (13) have designed a compound containing the stilbene group as the conjugated bridge, the organic boryl as acceptor and the crown ether group as donor, and could be identified by the fluorine anion by fluorescence emission change. In this paper, a series of novel stilbene derivatives with different donor-π-acceptor (D-π-A) structures were synthesized and polymerized via free radical polymerization. Their emissions were regulated by introducing the donor group to form the donor-π-acceptor (D-π-A) construction from green to orange and even red regions.

2 Experimental

2.1 Monomer Synthesis

A general route for the synthesis of three stilbene-type monomers is shown in Scheme 1.

2.1.1. *Synthesis of monomer 4-nitro-4'-methacrylate stilbene (NS)*

Benzoic acid benzaldehyde ester (I): A stirred mixture of 4-hydroxybenzaldehyde (12.2 g, 0.1 mol), distilled pyridine (9.48 g, 0.12 mol) and 50 ml of dry dichloromethane was treated dropwise with benzoyl chloride (15.4 g, 0.11mol) at ice-bath. The resulting solution was stirred at room temperature for 2 h and then washed with dilute hydrochloric and

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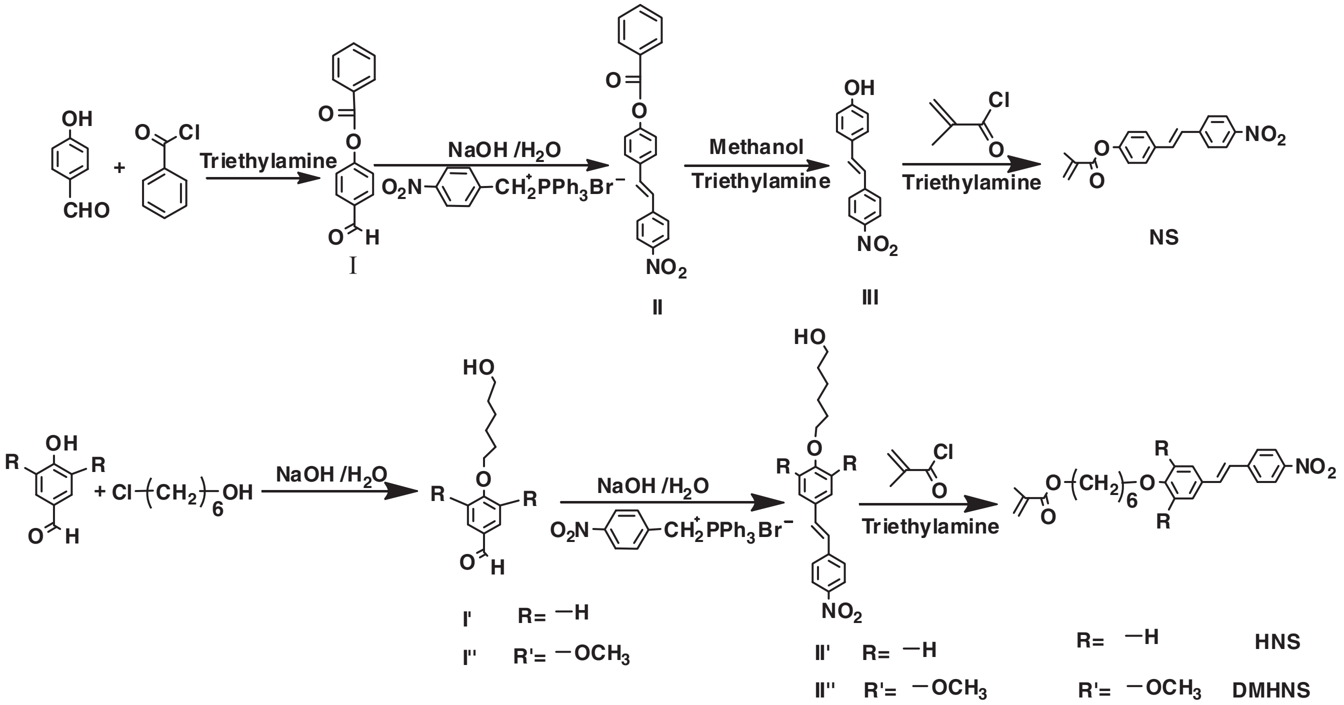


Sch. 1. Route for the synthesis of three stilbene-type monomers.

saturated sodium bicarbonate. The organic phase was separated, dried ($MgSO_4$) and evaporated to obtain a white solid (21.5 g, 95%). Element Anal. Cald. for $C_{14}H_{10}O_3$: C, 74.33%; H, 4.46%. Found: C, 74.21%; H, 4.51%. ^{1}H-NMR($CDCl_3$, 400 MHz) δ (ppm): 10.03(s, 1 H, –CHO, 8.21 (d, 2 H, –Ar), 7.98 (d, 2 H, –Ar), 7.68 (t, 1 H, –Ar), 7.54 (t, 2 H, –Ar), 7.43 (d, 2 H, –Ar).

(4-Nitrobenzyl)triphenylphosphonium Bromide: The detailed synthesis and characterization of compound was reported in reference (14). Element. Anal. Calcd. for $C_{25}H_{21}BrNO_2P$: C, 62.78%; H, 4.43%; N, 2.93%. Found: C, 62.71%; H, 4.52%; N, 2.87%. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ (ppm): 7.82 (m, 11 H, –Ar), 7.58 (m, 6 H, –Ar), 7.45 (d, 2 H, –Ar), 6.02 (d, 2 H, $-CH_2P$).

4-benzoyl-4′-nitro stilbene (II): (4-Nitrobenzfyl) triphenylphosphonium bromide (9.54 g, 0.02 mol), compound I (5.42, 0.024 mol) and a few crystals of tetrabutylammonium iodide were added to 50 ml dichloromethane, the concentration of 20% sodium hydroxide solution (50 ml) was poured into a stirred mixture. The mixture was stirred for 12 h and then the oil phase was separated, washed with deionized water three times, dried ($MgSO_4$), and the solvent was evaporated to give an yellow solid (2.97 g, 43%). Element Anal. Calcd. for $C_{21}H_{15}NO_4$: C, 73.03%; H, 4.38%; N, 4.06%. Found: C, 73.01%; H, 4.53%; N, 4.05%. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.20 (d, 2 H, –Ar), 8.12 (d, 2 H, –Ar), 7.64 (t,1 H, –Ar), 7.52 (t, 2 H, –Ar), 7.43 (d, 2 H, –Ar), 7.14 (d, 2 H, –Ar), 6.80 (d, 1 H, –CH=), 6.66 (d, 1 H, vinyl proton).

4-hydro-4′-nitro stilbene (III): Compound II (3 g,8.7 mmol) was suspended in a solution of methanol (200 ml) and triethylamine (9.09 g, 0.09 mol). The reaction mixture was refluxed for 24 h. Methanol was removed under reduced pressure then re-dissolved in ethyl acetate. The solution was washed with dilute hydrochloric and saturated sodium bicarbonate, dried with Mg_2SO_4, and evaporated to give an orange-red solid (1.78 g, 85%). Element Anal. Calcd. for $C_{14}H_{11}NO_3$: C, 69.70%; H, 4.60%; N, 5.81%. Found: C, 69.63%; H, 4.71%; N, 5.80%. ^{1}H-NMR (DMSO-d6, 400 MHz) δ: 8.18 (d, 2 H, –Ar), 7.80 (d, 2 H, –Ar), 7.54 (d,2 H, –Ar), 7.40 (d, 1 H, –CH=), 7.20 (d, 1 H, vinyl proton), 6.85 (d, 2 H, –Ar).

4-methylacrylate-4’-nitro stilbene (NS): A mixture of compound III(2 g, 6.5 mmol), triethylamine (0.78 g, 7.8 mmol) and 50 ml of dry THF was treated dropwise with methacryloyl chloride (0.74 g, 7.1 mmol) at ice-bath. The reaction mixture was poured into water, filtered, and recrystallized by ethanol to give monomer NS (1.63 g, 81%). Element Anal. Calcd. for $C_{18}H_{15}NO_4$: C, 69.89%; H, 4.89%; N, 4.53%. Found: C, 69.87%; H, 4.95%; N, 4.51%. ^{1}H-NMR ($CDCl_3$, 400MHz) δ: 8.23 (d, 2 H, –Ar), 7.64 (d, 2 H, –Ar), 7.65 (d, 2 H, –Ar), 7.24 (d, 1 H, –CH=), 7.18 (d, 2 H, –Ar), 7.12 (d, 1 H, vinyl proton), 6.34 (s, 1 H, vinyl proton in acrylate), 5.78 (s, 1 H, vinyl proton in acrylate), 2.08 (s, 3 H, $-CH_3$), m. p.: 165–167°C.

2.1.2. *Synthesis of monomer 4-(6-methacrylate)hexyl-4′-nitro stilbene (HNS)*

4-((6-Hydroxyhexyl)oxy)benzaldehyde (I’): The synthesis route of compound was listed in the reference(15). Element Anal. Calcd. for $C_{13}H_{18}O_3$: C, 70.24%; H, 8.1%. Found: C, 70.21%; H, 8.19%. ^{1}H-NMR($CDCl_3$,400 MHz)δ (ppm): ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 9.88 (s, 1 H, −CHO), 7.82 (d, 2 H, −Ar), 6.99 (d, 2 H, −Ar), 4.05 (t, 2 H, methylene proton adjacent to phenol), 3.68 (t, 2 H, methylene proton adjacent to hydroxy), 1.84 (m, 2 H, $-CH_2-$), 1.60 (m, 2 H, $-CH_2-$), 1.48 (m, 4 H, $-CH_2-$).

4-((6-Hydroxyhexyl)oxy)-4’-nitro stilbene (II’): The synthesis route of stilbene derivative III’ was similar with compound II. The yellow solid was passed through a silica column with ethyl acetate/petroleum ether = 1:1 (v/v) as developer, 23%. Element Anal. Calcd. for $C_{20}H_{23}NO_4$: C, 70.36%; H, 6.79%; N, 4.10%. Found: C, 70.33%; H, 6.89%; N, 4.12%. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.20 (d, 2 H, −Ar), 7.60 (d, 2 H, −Ar), 7.48 (d, 2 H, −Ar), 7.19 (d, 1 H, −CH=), 7.01 (d, 1 H, vinyl proton), 6.92 (d, 2 H, −Ar), 4.00 (t, 2 H, methylene proton adjacent to phenol), 3.67 (t, 2 H, methylene proton adjacent to −OH), 1.82 (m, 2 H, $-CH_2-$), 1.62 (m, 2 H, $-CH_2-$), 1.48 (m, 4 H, $-CH_2-$).

4-(6-methacrylate)hexyl-4’-nitro stilbene (HNS): The bright-yellow solid was obtained according to the syntheses route of NS, 79%. Element Anal. Calcd. for $C_{24}H_{27}NO_5$: C, 70.40%; H, 6.65%; N, 3.40%. Found: C, 70.29%; H, 6.76%; N, 3.35%. ^{1}H-NMR ($CDCl_3$, 400MHz) δ: 8.21 (d, 2 H,−Ar), 7.61 (d, 2 H, −Ar), 7.47 (d, 2 H, −Ar), 7.20 (d, 1 H, −CH=), 7.02 (d, 1 H, vinyl proton), 6.92 (d, 2 H, −Ar), 6.10 (s, 1 H, vinyl proton), 5.55 (s, 1 H, vinyl proton), 4.16 (t, 2 H, methylene proton), 4.00 (t, 2 H, methylene proton), 1.95 (s, 3 H, $-CH_3$), 1.82 (m, 2 H, $-CH_2-$), 1.72 (m, 2 H, $-CH_2-$), 1.51 (m, 4 H, $-CH_2-$), m. p.: 92–94°C.

2.1.3. *Synthesis of monomer 4-(6-methacrylate)hexyl-2,3-dimethoxy-4′-nitro stilbene (DMHNS)*

4-((6-Hydroxyhexyl)oxy)-3,5-dimethoxy benzaldehyde (I”): This compound was prepared according to the synthesis route of compound I’, 97%. Element Anal. Calcd. for $C_{13}H_{18}O_3$: C, 70.24%; H, 8.16%. Found: C, 70.21%; H, 8.19%. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 9.87 (s, 1 H, −CHO), 7.13 (s, 2 H, −Ar), 4.08 (t, 2 H, methylene proton), 3.92 (s, 6 H, $-OCH_3$), 3.66 (t, 2 H, methylene proton), 1.78 (m, 2 H, $-CH_2-$), 1.60 (m, 2 H, $-CH_2-$) 1.44 (m, 4 H, $-CH_2-$).

4-((6-Hydroxyhexyl)oxy)-3,5-dimethyl-4′-nitro stilbene (II”): The synthesis route of stilbene derivative II” was similar to 4-benzoyl-4’-nitro-stilbene (II). The salmon pink liquid was passed through a silica column with dichloromethane as a developer to obtain the crude product, which was used for the next reaction without further purification, 24%.

4-(6-methacrylate)hexyl-2,3-dimethoxy-4′-nitro stilbene (DMHNS): The salmon pink liquid was obtained according to the synthesis route of NS, passed through a silica column with ethyl acetate/ petroleum ether = 2:3 (v/v) as the developer, 79%. Element Anal. Calcd. for $C_{26}H_{31}NO_7$: C, 66.51%; H, 6.65%; N, 2.98%. Found: C, 66.46%; H, 6.73%; N, 2.94%. ^{1}H-NMR ($CDCl_3$, 400MHz) δ: 8.22, 8.10 (d, 2 H, −Ar); 7.62, 7.44 (d, 2 H, −Ar); 7.18, 7.06 (d, 2 H, −Ar); 6.77, 6.14 (s, 1 H, −Ar); 6.70, 6.58 (d, 1 H, −CH=); 6.09 (s, 1 H, −Ar); 5.54 (s, 1 H, vinyl proton), 4.15 (t, 2 H, methylene proton); 4.00 (t, 2 H, methylene proton); 3.90, 3.64 (s, 6 H, $-OCH_3$); 1.93 (s, 3 H, $-CH_3$); 1.78 (m, 2 H, $-CH_2-$); 1.70 (m, 2 H, $-CH_2-$); 1.53 (m, 4 H, $-CH_2-$). M.p.: liquid in −18°C .

2.2 Polymerization

The stilbene side-chain polymers (NS, HNS and DMHNS) were prepared by a free radical method using AIBN as an initiator, shown in Scheme 2. The detailed reaction conditions and typical polymerization procedure are as follows. The monomer (0.95 mmol) and 7.8 mg (5 mol% to the monomer) of AIBN were dissolved in cyclohexanone (2.0 ml). The solution was thoroughly degassed by several freeze-pump-thaw cycles and heated in a sealed ampoule at 100°C for 12 h. The resulting polymer solution was cooled and poured into methanol. The precipitated polymer was filtered and dissolved in tetrahydrofuran, and precipitated into methanol several times in order to remove the excessive monomers.

2.3 Instruments

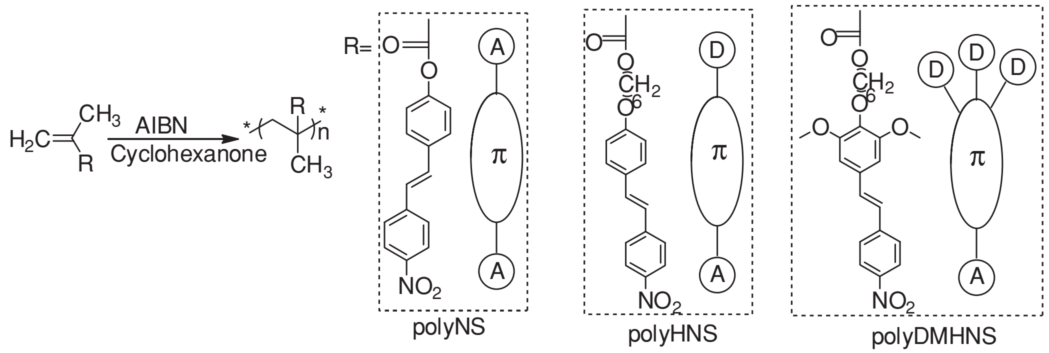
The elemental analysis was performed by an Italian 1106FT analyzer. ^{1}H-NMR spectra were obtained on an Inova 400 MHz spectrometer using $CDCl_3$ as a solvent. Gel permeation chromatography (GPC) analysis was carried out on a Waters 1515 chromatograph equipped with a refractive index detector, THF as an eluent, and calibrated with polymethyl methacrylate at standards. The UV-Vis absorption spectra were measured with a UV-3150 spectrophotometer. The fluorescent spectra were obtained at room temperature with Edinburgh-920 spectrophotometer.

3 Results and Discussion

3.1 Synthesis and Characterization

The basic synthetic routes of three monomers were presented in Scheme 1. The structure of intermediate products and monomers were characterized with elemental analysis and ^{1}H-NMR data, which were in good agreement.

The polymerization of these stilbene-monomers was carried out using AIBN as an initiator. Their structures were confirmed by ^{1}H-NMR spectra. As shown in Figure 1, the chemical shifts assigned to the vinyl protons of three monomers disappeared after the polymerization. A novel chemical shift of $-CH_2-$ was observed at $\delta = 0.8$–2.0 ppm



Sch. 2. The synthetic route and the structure of stilbene monomers.

in all polymers. The signals at 3.7-4.2 ppm originated from the protons of $-CH_2-$ in polyHNS (labeled as a and b), and those at 3.4-4.2 ppm were attributed to the $-CH_2-$ protons in polyDMHNS (labeled as a, b and c).

Table 1 shows the number average molecular weight (M_n, GPC) and polydispersity index (PDI) of polymers with different stilbene-type side-chain. The number average molecular weights of polymers ranged from 4900 to 10300 with the conversion rate located at 42–57%, and the polydispersity index is about the vicinity of 1.6. According to the GPC result, the degree of polymerization is limited due to inhibition effect of the nitro group (16). PolyNS has poor solubility at non-polar and weak polar organic solvents such as toluene and chloroform and only dissolves in a strong polar solvent such as DMF and DMSO. However, solubility of PolyHNS and PolyDMHNS was improved in the organic solution by introducing the donor moieties to the system.

3.2 Optical Properties

The UV-Vis spectra of monomers and polymers in DMF and THF solution were investigated as shown in Table 2. The monomers and their accordingly polymers show similar absorption pattern and solvent polarity have no obvious effect on their absorption.

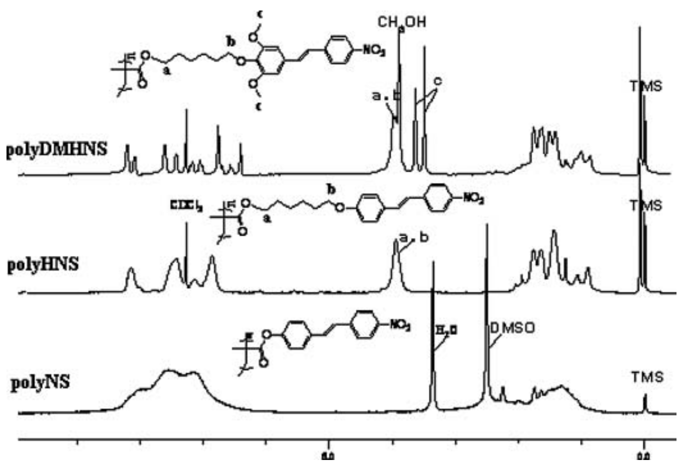


Fig. 1. ^{1}H-NMR spectra of three polymers (polyNS in DMSO, polyHNS and polyDMHNS in $CDCl_3$).

Figure 2 shows the UV-Vis spectra of three stilbene-type polymers in DMF solution. An intense characteristic absorption band originating from the $\pi - \pi^*$ transition of three chromophores was observed, but the position is strongly affected by the structure of the chromophore. For example, compared with that of polyNS ($\lambda_{\max} =$ 353 nm), the $\pi - \pi^*$ transitions of polyHNS ($\lambda_{max} = 378$ nm) and polyDMHNS ($\lambda_{\max} =$ 381 nm) are obviously red-shifted by introducing hexyloxy (polyHNS) and auxochrome dimethyloxy (polyDMHNS) as donor group, respectively (17). It indicates strong intermolecular charge transfer (ICT) from the donor group hexyloxy to the acceptor group nitro was occurred in polyHNS and polyDMHNS.

The fluorescent emission spectra of these monomers and polymers were also investigated (Table 2). The emission bands of monomer and accordingly, the polymers are similar. The band at 541 nm (green region) is the emission of NS and polyNS originated from the A-π-A structure; the peak at 610 nm is the emission of HNS and polyHNS due to the D-π-A structure; the band at 680 nm (red region) originated from the D'-π-A structure of DMHNS and polyDMHNS. These obvious emission shifts from green to red is mainly ascribed to the effective donor-acceptor structure.

Table 1. The polymerization results of the stilbene-containing monomers

Polymer	*Temperature (°)*	*Time (h)*	*Yield (%)*	*GPC*^{a} M_n	*GPC*^{a} M_w/M_n
polyNS	100	12	42	4968	1.628
polyHNS			57	6885	1.624
polyDMHNS			54	10238	1.620

[a] Molecular weights determined by GPC using polymethyl methacrylate as the standards.

Table 2. UV–Vis ($\lambda_{ab\ max}$), Fl spectra ($\lambda_{em\ max}$) and fluorescence quantum yield (Φ_f)of three stilbene-type monomers and polymers in different solvents

	UV-Vis		*FL*		
	THF	*DMF*	*THF*	*DMF*	Φ_f
NS (nm)[a]	355	357	506	541	8.5×10^{-3}
polyNS (nm)[a]	349	353	507	543	8.3×10^{-3}
HNS (nm)[b]	377	384	526	616	0.21
polyHNS (nm)[b]	375	383	528	609	0.064
DMHNS (nm)[c]	377	382	596	703	0.027
PolyDMHNS (nm)[c]	378	381	598	682	0.021

[a] $c = 10 \times 10^{-5}$ M, $\lambda_{ex} = 350$; [b] $c = 5 \times 10^{-5}$ M, $\lambda_{ex} = 375$; [c] $c = 5 \times 10^{-5}$ M, $\lambda_{ex} = 377$ nm.

However, the emission is sensitive to the polarity of solvent, and exhibits bathochromic shifts with increasing polarity of the solvent. For example, both the emission of monomer NS and its polymer PolyNS were located at about 506 nm in THF solution and red-shifted to 541 nm in DMF solution, which is ascribed to its A-π-A structure and the electrostatic gravitation between the chromophore and solution. More red-shift values could be found in emission of HNS and PolyHNS ($\lambda_{em} = 526$ nm in THF) and there's at least 90 nm shift in DMF solution ($\lambda_{em} = 616$ nm); emission of DMHNS and PolyDMHNS was located at 596 nm in THF solution, and more than 80 nm red-shift found in DMF. The dramatic red-shift emission of HNS, DMHNS and their respective polymers could be attributed to their D-π-A structure and the intramolecular charge transfer process (ICT) between the nitro group (as an electron acceptor)

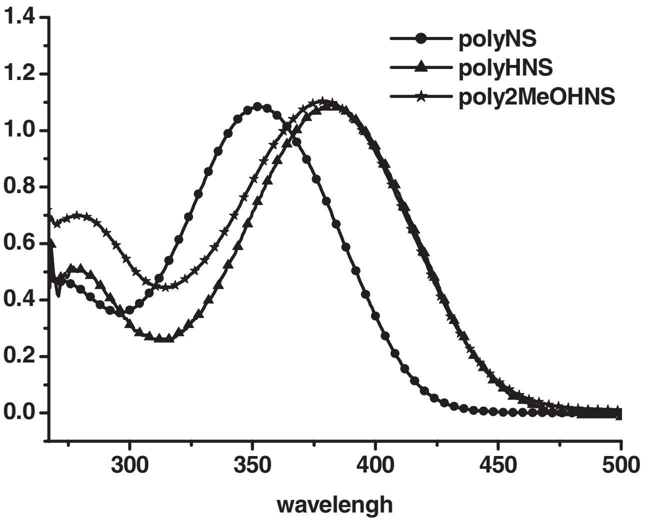
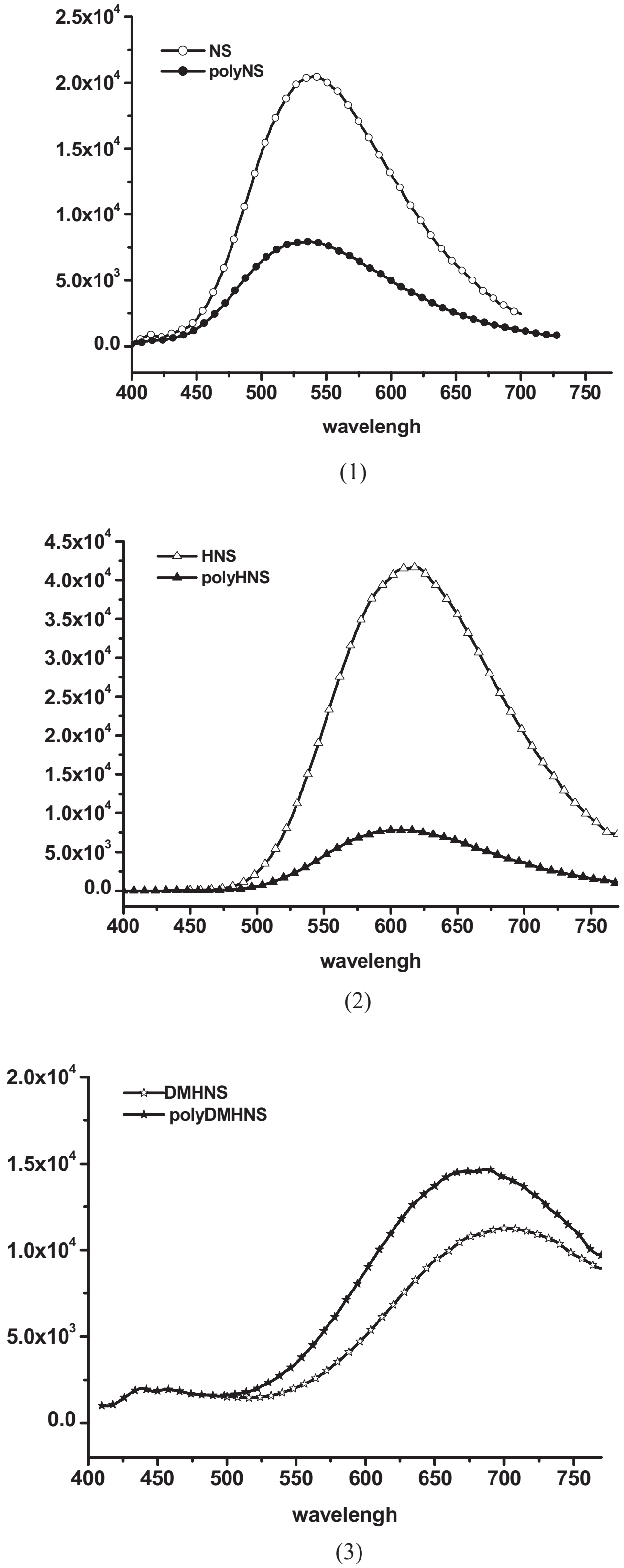


Fig. 2. UV-Vis spectra of three stilbene-polymers in DMF solution.

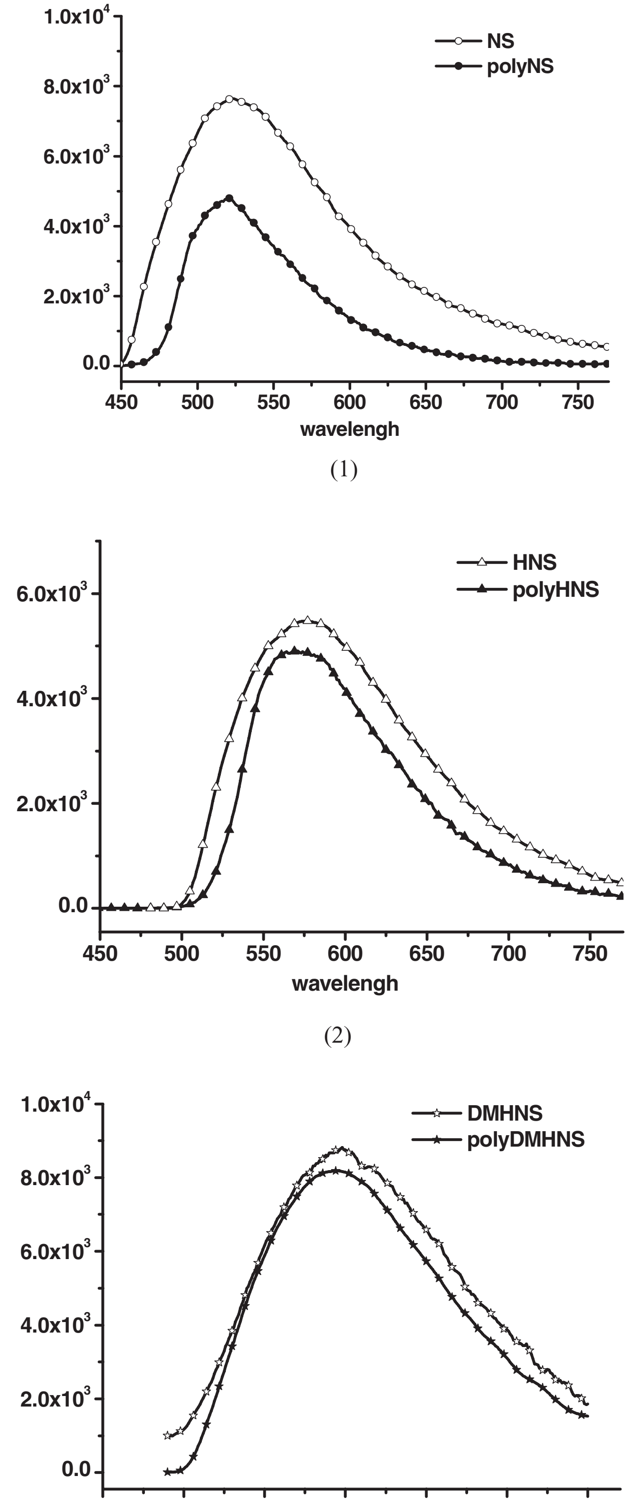


(1)

(2)

(3)

Fig. 3. Fluorescent spectra of stilbene contained monomers and polymers in DMF solution ($c = 10^{-5}$M, $\lambda_{ex} = 350$ nm for NS and PolyNS (1), 375 nm for HNS and PolyHNS (2) and 377 nm for 2MeOHNS and PolyDMHNS (3), respectively.



(1)

(2)

(3)

Fig. 4. Fluorescent spectra of stilbene contained monomers and polymers in solid state (with $\lambda_{ex} = 360$ nm for NS and PolyNS (1), 380 nm for HNS and PolyHNS and 420 nm for 2MeOHNS (2) and PolyDMHNS (3), respectively).

and the hexyloxy or dimethoxyl (as electron donor) (18). The fluorescence quantum yields for these monomers and polymers in DMF solution have been determined in Table 2. Compared with NS and PolyNS, the increase of Φ_f in monomers HNS, DMHNS and their monopolymers can be attributed to the D-π-A structure which partly prevented photo-isomerization quenching of the fluorescence (19). The introduction of flexible group, methyloxy in DMHNS may decrease the molecular planarity and the monomer DMHNS shows lower Φ_f than that of HNS. However, the decrease of Φ_f in polymers is attributed to more vibrations of *cis-trans* isomers occurred in soft polymer chain.

The fluorescent properties of stilbene containing monomers and polymers may show some interesting emission tuning by introducing hexyloxy and dimethoxy to form the D-π-A structure. The fluorescence spectra of these monomers and polymers in DMF solution are shown in Figure 3.

Figure 4 shows the emission spectra of three stilbene containing monomers and polymers in solid state, which are different from that in DMF solution. For example, the emission of NS and polyNS is located at 524 nm in the green region. When introducing hexyloxy moieties to form a D-π-A structure with nitro group, the emission of HNS and polyHNS are red-shifted 54 nm to the yellow region; when the strength of the donor group increased, 66 nm red-shifted for DMHNS and polyDMHNS compared to chromophore NS was observed. The different emission in DMF solution and film is possibly attributed to a different aggregating state. In addition, the fluorescent intensities of three polymers are obvious lower than that of their monomers whether in solution or in solid state. It can be attributed to the possibility of a chromophore congregation.

4 Conclusions

A series of novel acrylate monomers containing stilbene derivative moiety (NS, HNS and DMHNS) were synthesized by a Witting reaction and polymerized by free radical polymerization. The emission of three stilbene type monomers and polymers was sensitive to different solvents and the emission band showed dramatic red-shifts with increasing polarity of the solvent. Meanwhile, the fluorescent emission is tunable from green to orange and red regions by introducing the donor group to form a D-π-A structure or by increasing the strength of the donor group both in DMF solution and in solid state. The monomers can also copolymerize with other functional monomers to obtain functional polymers applied, such as a fluorescent probe. This job is ongoing in our lab.

Acknowledgements

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