Synthesis of Polyimide Oligomer Attached with Hemicyanine Dye

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ABSTRACT: Two hemicyanine dyes, trans-4-[p-(N,N-di(2-hydroxyethyl)) amino-styryl]-N-methylpyridinium tetraphenylborate (dye- C_1) and trans-4-[p-(N,N-di(2-hydroxyethyl)) amino-styryl]-N-octylpyridinium tetraphenylborate (dye- C_8) were synthesized, and characterized by infrared, ¹H-NMR, thermoanalysis, respectively. Then polyimide (PI) oligomers (referred to as P- C_1 and P- C_8) with these hemicyanine dyes attached to the polymer side chain were pre-

pared through Mitsunobu condensation. When the linear optical properties of the two dyes and the two PI oligomers were studied in dimethylformamide solvent, there were obvious blue shifts in the spectra of the oligomers. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 1448–1453, 2009

Key words: hemicyanine dye; fluorescence; polyimide; oligomer

INTRODUCTION

Today, many studies have been devoted to the synthesis and characterization of fluorescent polymers, such as poly(1,4-phenylenevinylene),^{1,2} polyalkyl fluorene,^{3,4} poly(*p*-phenylene),^{5,6} polythiophene derivatives,^{7,8} and polyimide (PI),⁹⁻¹⁶ because these polymers could be used as luminescent materials. For practical applications, these polymeric materials must retain high optical quality as thin films and the feasibility of device fabrication. Among them, PI materials have been widely used in microelectronic devices because of their excellent thermomechanical properties, low dielectric constants, and high dissipation factors. Generally, the optically active chromophores were incorporated either by doping or by attaching via covalent bonds to the polymer backbone. Most published synthesis methods included the polymerization of polyamic acid precursors and imidization to give the cyclic imide structure, but chemical conditions of the monomer synthesis and the imidization of the polymer were relatively harsh and few chromophores could survive it. Yoon and Shim¹¹ have reported a facile approach for the synthesis of optically functionalized PI, which is a direct preparation of PI from diimide monomer and dihydroxy mono-

mer through the Mitsunobu condensation, this process certified that the chromophore could be incorporated in the PI backbone without the decomposition of its molecules. Among numerous chromophores, amino-styrylpyridinium salts (hemicyanine dyes) have found some successful applications in science and technology, and have been commonly applied to lasers, electronics, and nonlinear optics.¹⁷⁻ Moreover, efforts to the synthesis and application of some hemicyanine dyes, which possess a donor group on one end and an acceptor group on the other end and have excellent single-photon and two-photon optical properties have been also devoted.22-25 Although our compounds, trans-4-[p-(N, N-hydroxyethyl) amino-styryl]-N-methylpyridinium iodide (DHEASPI- C_1) and trans-4-[p-(N, N-hydroxyethyl) amino-styryl]-N-octylpyridinium bromide (DHEASPBr-C₈), they possess strong TPA and linear optical properties because they have highly conjugated cations (*i.e.*, DHEASP- C_1^+ and DHEASP- C_8^+),²⁴ both dyes are poorly soluble in normal organic solvents (such as tetrahydrofuran (THF) and acetone), so synthesis of another two hemicyanine dyes (*i.e.*, dye- C_1 and dye- C_8) by replacing the iodide ion (I⁻) and bromide ion (Br⁻) with tetraphenylborate ion (BPh₄⁻) were put under investigation in this research, where the synthesis of a PI containing this kind of hemicyanine dye through the Mitsunobu condensation under facile conditions was performed.

The chemical structures of the hemicyanine dyes are shown in Scheme 1, and that of DHEASPI- C_1 and DHEASPBr- C_8 are also presented for comparison, where the four dyes possess an asymmetric molecule

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Scheme 1 Molecular structures of DHEASPI- C_1 , DHEASPB- C_8 , dye- C_1 and dye- C_8 .

structure, consisting of different terminal substituted pyridinium groups as acceptors and the *N*-phenyldiethanolamine group as the donor and their solubilities were different. Thus, a comparative study of the linear absorption properties and single-photon fluorescence behavior of all the samples is reported to understand the influence of the dye cation and anion on the optical properties and the oligomer coupling of the dihydroxy group in the dye with diimide compounds through Mitsunobu condensation were performed.

EXPERIMENTAL

Materials

N-phenyldiethanolamine was purchased from Acros Organic and used without further purification. Other solvents (A.R.) and reagents were obtained commercially and used without further purification except for dimethylformamide (DMF), ethanol and methanol.

DHEASPI-C₁ and DHEASPBr-C₈, as substrates for the synthesis of dye-C₁ and dye-C₈, respectively, were synthesized.²⁴

Measurements

The ¹H-NMR spectra were recorded with the use of a GCT-TOF NMR spectrometer at 400 MHz. Dimethylsulfoxide (DMSO) and deuterated chloroform (CDCl₃) were used as the solvents, respectively, and tetramethylsilane as the internal standard.

Infrared (IR) spectra were measured on a Nicolet 5200 Fourier transform infrared (FT-IR) 5DX instrument using solid samples dispersed in KBr pellets.

The melting points and decomposition temperatures were measured on a Perkin-Elmer Diamond 5700 thermogravimetric analyzer at a heating rate of 20°C/min under nitrogen atmosphere.

UV–vis absorption spectra were recorded at room temperature in quartz cells of 1 cm path length using a TU-1800 SPC spectrophotometer, and then single-photon fluorescence spectra were obtained in the same cell under excitation of the respective maximum absorption wavelength on an Edinburgh 920 spectrofluorometer. In this article, all of the concentrations of the dye solutions in the linear spectra were 1.0×10^{-5} M, as no aggregation or self-absorption effects of the dyes were observed at this concentration. Then, the fluorescence quantum yield (Φ) of the dye was calculated in Ref. 24.

Synthesis of hemicyanine dyes

The synthetic procedure is shown in Scheme 2. The structure and purity of the prepared compounds were confirmed by ¹H-NMR spectroscopy and IR spectra or elemental analysis.

Trans-4-[*p*-(*N*,*N*-di(2-hydroxyethyl))amino-styryl]-*N*-methylpyridinium tetraphenylborate (dye-C₁)

A mixture of 4-(*N*,*N*-diethanolamino) benzaldehyde (0.05 mol, 10.5 g) and 4-methyl-*N*-methylpyridinium iodide (0.05 mol, 11.75 g) was stirred in methanol (300 mL) at reflux temperature overnight in the presence of piperidine (a few drops). Then the solvent was removed and the red crude solid was purified by column chromatography on silica gel with methanol as eluent.²⁴ DHEASPI-C₁ was obtained with yield 71% and T_d 306.5°C.

To a solution of DHEASPI-C₁ (0.03 mol, 12.8 g) in methanol (200 mL) was added dropwise to a solution of sodium tetraphenylborate (10.3 g, 0.03 mol) in methanol (100 mL). Several hours later, a red precipitate was formed. After cooling, the precipitate was washed with methanol several times, collected



Scheme 2 A general route for the synthesis of dye- C_1 and dye- C_8 .

by filtration, and then dried. Yield 67% and T_m 182.6°C. ¹H-NMR (DMSO-d₆, 400 MHz) δ : 8.66 (2H, Py—CH, d, J 6.4 Hz), 8.02 (2H, Py—CH, d, J 6.4 Hz), 7.87 (1H, CH=CH, d, J 16.0 Hz), 7.56 (2H, Ar—H, d, J 8.8 Hz), 7.18 (20H, Ar—H, q), 7.11 (1H, CH=CH, d, J 16.4 Hz), 6.80 (2H, Ar—H, d, J 6.0 Hz), 3.54 (8H, —CH₂—, q). Elemental analysis: Calcd C, 81.55; H, 7.01; N, 4.53. Found: C, 81.08; H, 7.38; N, 4.42.

Trans-4-[*p*-(*N*,*N*-di(2-hydroxyethyl))amino-styryl]-*N*-octylpyridinium tetraphenylborate (dye-C₈)

The synthetic procedure for dye-C₈ was similar to that for dye-C₁. Yield 56% and T_m 48.3°C. ¹H-NMR (DMSO-d₆, 400 MHz) δ : 8.75 (2H, Py–CH, d, J 6.0 Hz), 8.07 (2H, Py–CH, d, J 6.4 Hz), 7.91 (1H, CH=CH, d, J 19.2 Hz), 7.60 (2H, Ar–H, d, J 7.6 Hz), 7.52 (20H, Ar–H, q), 7.35 (1H, CH=CH, d, J 13.2 Hz), 6.83 (2H, Ar–H, d, J 8.8 Hz), 4.28 (4H, –CH₂–, t, J 5.8 Hz), 3.71 (4H, –CH₂–, t, J 5.8 Hz), 1.57 (2H, –CH₂–, m), 1.25 (12H, –CH₂–, m), 0.88 (3H, –CH₃, t, J 6.2 Hz). Elemental analysis: Calcd C, 82.10; H, 8.02; N, 3.91. Found: C, 81.95; H, 7.94; N, 3.67.

Synthesis of pyromellitic diimide

A total of 4.36 g (0.02 mol) pyromellitic dianhydride (PMDA) was reacted with 2.4 g (0.04 mol) of urea at 200°C for 3 h with anhydrous Al_2O_3 powder but no solvent, and then cooled to room temperature. The reaction mixture was washed with distilled water several times and dried thoroughly. After that, the neat mixture of the diimide and Al_2O_3 powder was dissolved in DMF solvent and white diimide crystals were formed in the concentrated filtrate. Yield 60% and T_d 331.5°C. IR (KBr pellet, cm⁻¹): 1771.4 and 11,699.1 (C=O and CO–NH), ¹H-NMR (DMSO-d₆, 400 MHz) δ : 11.82 (2H, CO–NH, s), 8.06 (2H, Ar–H, s). Elemental analysis: Calcd C, 55.57; H, 1.87; N, 12.96. Found: C, 55.60; H, 1.83; N, 12.73.

Synthesis of PI oligomers (P-C₁ and P-C₈)

To a mixture of 2.16 g (0.01 mol) pyromellitic diimide and quantitative (0.01 mol) dye- C_n (n = 1 or 8) in 50 mL of anhydrous THF, 3 equiv (0.03 mol) of triphenylphosphine (PPh₃) was added. Then 3 equiv (0.03 mol) of diethyl azodicarboxylate (DEAD) was added gradually into that mixture. The mixture's color changed to dark red and the polymerization reaction was complete after 10 h. Finally, the polymer mixture was poured into ice H₂O/CH₃OH (1 : 1 volume) and the precipitate was collected and washed with blending solvent several times.

The polymerization reaction between the diimide and dihydroxy monomers containing the hemicyanine moiety was executed by the Mitsunobu reaction using DEAD and PPh₃ in THF solvent. After polymerization, the peak at 11.82 ppm, which corresponds to the imide proton of the monomer disappeared completely in the final polymer, indicating the reaction between diimide and dihydroxy monomer. In the case of P-C₈, a similar condition was observed.

RESULTS AND DISCUSSION

Characterization of dyes

The ¹H-NMR spectra of the two dyes displayed two characteristic doublets localized at chemical shifts of about 7 and 8 ppm. They were both attributed to vinyl hydrogen atoms. Based on the large coupling constant for the olefinic proton (J = 16-19), it was concluded that these dyes existed as the all-trans conformation in the ground state.²⁶

Solubility of different dyes

Table I shows the different solubility of the dyes. When the anion was Br^- and I^- , the dye could dissolve in ethanol and H_2O , but not in THF and acetone. On the other hand, when the anion was $B(Ph_4)^-$, the dye could dissolve in THF and acetone, but not in ethanol and H_2O . So, the anion in the dyes had a great influence on solubility, and dyes with $B(Ph_4)^-$ were easy to dissolve in organic solvent, which gave an advantage for further polymerization.

Synthesis of pyromellitic diimide and PI oligomers $(P-C_1 \text{ and } P-C_8)$

The synthesis of the diimide monomer from the dianhydride was a one-pot imidization of PMDA using urea as the nitrogen source. The synthetic procedure is shown in Scheme 3. The anhydride compounds were reacted with urea at 200°C to give nucleophilic substitution of nitrogen into the anhydride moiety. After the completion of the reaction, the peaks corresponding to the imide protons were observed at 11.82 ppm in the ¹H-NMR spectrum.

TABLE ISolubility of the Four Different Dyes

Solvent	THF	Acetone	Ethanol	DMF	CH ₃ CN	H ₂ O
DHEASPI-C ₁	_	_	± +	+	+	+
Dye-C ₁	+	+	— —	+	+	- -
Dye-C ₈	+	+	±	+	+	-

+ Means solvable, \pm Means solvable difficultly, –Means insolvable.



Scheme 3 Synthetic pathway for $P-C_1$ and $P-C_8$.

The molecular weights (M_n and M_w) of the PI oligomers are shown in Table II and the values are not high. The bulkiness and ionic character of the tetraphenylborate anion should have probably retarded the Mitsunobu reaction to give a shorter polymer chain.

The PI oligomer structures were characterized by FT-IR spectroscopy. FT-IR spectra of the PI oligomer (P-C₁) showed the characteristic absorption peak at 1720 cm⁻¹ attributed to the imide rings, which did not appear in the spectrum of dye-C₁. P-C₈ showed a similar peak at 1729 cm⁻¹.^{12,13}

Linear absorption and single-photon fluorescence spectroscopic analyses

In Figure 1 and Table III, one can see that the fluorescence spectra are inclined to red shift as the solvent dipole moment ($\mu/10^{-30}$ C.m²⁷) increases. That are like trans-4-(*p*-Pyrrolidinostyryl)-*N*-methylpyridinium iodide (PSPI) mentioned before, its predicted dipole moments obtained by PM3 calculations confirm that the dipole moment of PSPI in the excited state is greater than that in the ground state by 3.4 D.²³ The solvatochromic shifts were processed by applying the Lippert–Mataga equation.²⁸

TABLE II Physicochemical Properties of the Two Polyimide Oligomers

Oligomer	M_n	M_w	MDI	$\begin{array}{c} \lambda_{abs} \\ \left(nm \right)^a \end{array}$	$\begin{array}{c} \lambda_{em} \\ (nm)^a \end{array}$	Φ^{b}	$T_d/^{\circ}C^{\circ}$
P-C ₁	1491	2180	1.46	438	551	0.0483	234.0
P-C ₈	1772	2375	1.34	435	556	0.0281	219.3

 $^{\rm a}$ λ Is the peak location of maximum value in the spectrum and solvent is DMF.

^b Φ is fluorescence quantum yield.

^c The temperature where weight remained up to 95%.

$$hc(v_{abs} - v_{em}) = \frac{1}{4\pi\varepsilon_0} \frac{2}{\alpha^3} \mu_e \left(\mu_e - \mu_g\right) \Delta f + \text{const};$$
$$\Delta f = \frac{\varepsilon - 1}{2\varepsilon + 1} - \frac{(n^2 - 1)}{(2n^2 + 1)}$$

where μ_e and μ_g are the dipole moments in the excited and ground state, respectively, $(v_{abs}-v_{em})$ is the Stokes shift, ε is the dielectric constant, *n* is the optical refractive index, and Δf is the solvent polarity function.



Figure 1 Linear absorption (solid) and single-photon fluorescence spectra (dash) of samples in DMF solvent at $d_0 = 1 \times 10^{-5} \text{ mol/L}$ ((a) DHEASPI-C₁; (b) DHEASPBr-C₈; (c) Dye-C₁; (d) Dye-C₈). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley. com.]

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		at $u_0 = 1 \times 10$	mon/L an					
Solvent $\mu/10^{-30}$ C.m Δf		Ethyl acetate	THF	DMF	Acetone	Ethanol	CH ₃ CN	H ₂ O
		6.27	5.70	12.88	8.97	5.60	11.47	6.47
		0.1996	0.2095	0.2752	0.2852	0.2880	0.3054	0.3201
Dye-C ₁	λ_{abs} (nm)	473	480	470	471	/	468	/
	λ_{em} (nm)	594	601	606	600	/	605	/
	v_{abs} - v_{em} (cm ⁻¹)	4306.6	4194.4	4775.0	4564.8	/	4838.6	/
Dye-C ₈	λ_{abs} (nm)	469	476	466	466	/	464	/
	λ_{em} (nm)	592	597	601	599	/	598	/
	v_{abs} - v_{em} (cm ⁻¹)	4430.1	4258.0	4764.7	4820.3	/	4829.3	/
DHEASPI-C ₁	λ_{abs} (nm)	/	/	467	/	472	465	452
	λ_{em} (nm)	/	/	606	/	596	602	595
	v_{abs} - v_{em} (cm ⁻¹)	/	/	4911.6	/	4407.9	4894.1	5317.2
DHEASPB r-C ₈	λ_{abs} (nm)	/	/	479	/	484	475	456
	λ_{em} (nm)	/	/	613	/	602	608	599
	v_{abs} - v_{em} (cm ⁻¹)	/	/	4563.6	/	4049.9	4605.3	5235.3

TABLE III Spectral Values of Dye-C₁, Dye-C₈, DHEASPI-C₁, DHEASPBr-C₈, and in Different Solvents at $d_0 = 1 \times 10^{-5}$ mol/L and Δf of Different Solvents

Based on Table III and Ref. 24, the Lippert–Mataga plots for DHEASPI-C₁, DHEASPBr-C₈, dye-C₁, and dye-C₈ were made in Figure 2. It was noted that the four slopes were all greater than zero, so μ_e values were larger than μ_g . As a result, the fluorescence spectra of the dyes were inclined to red shift with the increase in the solvent dipole moment (μ).

For the comparison of the different anions, the slopes were calculated and one could see that the solvent



Figure 2 Plots of the Stokes shifts of different dyes vs. the solvent polarity function Δf (*det f*). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

effects in dye- C_1 and dye- C_8 were weaker than those in DHEASPI- C_1 and DHEASPBr- C_8 . This might be attributed to the special structure of BPh₄⁻ and lower dipole moment gaps (μ_e - μ_g). But it was also clear that the decrease in slope of dye- C_1 was larger than that of dye- C_8 , and the reason was probably because the heavy atom effect of I⁻ was more obvious than Br⁻.

In the UV spectra and fluorescence spectra, one could easily see that there were some blue shifts of characteristic peaks that corresponded to the two hemicyanine dyes (shown in Figure 3, Tables II and III). For example, the absorption maxima for the π – π * transition of the hemicyanine dye in dye-C₁ and P-C₁ were at 470 and 438 nm in DMF solvent, respectively, whereas that in dye-C₈ and P-C₈ were at 466 and 435 nm, respectively. When the monomer dye was introduced into the PI oligomer chain, the absorption maximum showed a blue shift of about 30 nm. This UV absorption pattern indicated that electronic interaction between the polymer chain and hemicyanine



Figure 3 Linear absorption (left) and single-photon fluorescence spectra (right) of the two oligomers in DMF solvent at $d_0 = 1 \times 10^{-5}$ mol/L. [Color figure can be viewed in the online issue, which is available at www. interscience.wiley.com.]



Figure 4 TGA scan spectrum for $P-C_8$ (heating rate $20^{\circ}C/\min$, N₂). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

dye occurred in the polymer matrix. There were similar phenomena in single-photon fluorescence spectra and the blue shift was about 50 nm.

Thermal properties of P-C₁ and P-C₈

The inherent thermal stability of P-C₈ is represented by the thermogravimetric analysis (TGA) curve presented in Figure 4 and Table II. The oligomer revealed an onset of weight loss at 219.3°C. The first weight loss corresponds to the decomposition of the tetraphenylborate group. The corresponding temperature of P-C₁ was at 234.0°C (Table II).

CONCLUSIONS

Two hemicyanine dyes, named dye- C_1 and dye- C_8 , were synthesized successfully and their optical properties have been studied in different solvents. When such hemicyanine dye substituted PI oligomers (P- C_1 and P- C_8) were prepared, these oligomers displayed similar characteristic absorption and fluorescence properties as well as excellent thermal stability.

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