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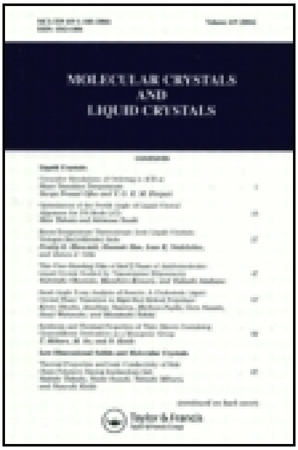

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Synthesis and Photolithographic Property of Conjugated Polymers with Polyazomethine Structure

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Synthesis and Photolithographic Property of Conjugated Polymers with Polyazomethine Structure

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Two different polyazomethine-type conjugated polymers, poly(phenoxiazine-3, 3′-dihydroxybensidine)(PZ-DHB) and poly(phenoxiazine-2,4-diamino-6-hydroxy-pyrimidine)(PZ-DHP) containing phenothiazine moiety and azomethine linkage in the main chain were synthesized by Schiff-base polycondensation reactions. The polymer poly(PZ-DHB) was converted to the photosensitive type, through Mitsunobu reaction by incorporation of a photocrosslinkable group, hydroxy-hexyloxystyryl-2-pyridine(2-HHSP) into the polymer side chains. The photosensitive conjugated polymer, poly(PZ-DHB)-PS, exhibited both strong fluorescence emission at 555 nm and good patterning property in the standard photolithographic processes.

Keywords: conjugated polymers; photolithography; polyazomethine

INTRODUCTION

Conjugated polymers have attracted much interest from researchers for their potential applications to the large area organic light emitting diodes (OLEDs) since the first polymer light-emitting diode (PLED) using poly(*p*-phenylenevinylene) (PPV) was reported by Friend et al. in 1990 [1–3]. Conjugated polymers can be easily converted to thin film

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by spin-coating process and the resulting PLED devices have merits of low threshold voltages and high external quantum efficiency. The PLEDs employing conjugated polymers, however, need to increase luminance efficiency and color purity compared with OLEDs containing small molecular light emitting materials.

Conjugated polymers such as PPV and polyfluorenes have been widely used in the fabrication of PLEDs [4–7]. Conjugated polymers containing heterocyclic structures have also been studied. For example polyazomethines with cyano groups were reported to have high luminance efficiency, thermal stability and good solubility to common organic solvents [8–11]. Weaver *et al.* reported that poly(1,4-phenylenemethylidynenitrilo-1,4-phenylenenitrinilomethylidyne) containing azomethine unit in the main chain exhibited similar performance as PPV in PLEDs in which the emitting layer was formed by chemical vapor deposition (CVD) method [10]. Fischer *et al.* also formed oligo-azomethine thin film utilizing aromatic diamine and dialdehyde monomers under high vacuum and found that the materials had good electron transfer properties [11]. In these works polyazomethine thin films were formed by thermal evaporation under high vacuum conditions.

In this work we synthesized heterocyclic conjugated polymers with azomethine units in the main chain and a photosensitive version of azomethine polymer. These polymers were investigated for their electroluminescent property and for the photolithographic process properties.

EXPERIMENTAL

1. Materials

Phenothiazine, 2-ethylhexyl bromide, phosphorus oxychloride, p-hydroxybenzaldehyde, 6-bromo-1-hexanol, sodium acetate, 2-picoline and 2,4-diamino-6-hydroxypyrimidine were purchased from Aldrich Chemical Co. and used as received. 3,3′-Dihydroxybenzidine, triphenylphosphine(PPh3) and diethylazodicarboxylate (DEAD) were purchased from TCI and used without further purification. Dimethylsulfoxide, dioxane, dimethyl formamide, 1,2-dichloroethane and other solvents were distilled before use.

2. Synthesis of Electroluminescent (EL) Polymers

Synthesis of intermediates and conjugated polymers with electroluminescent (EL) property are shown in Scheme 1.

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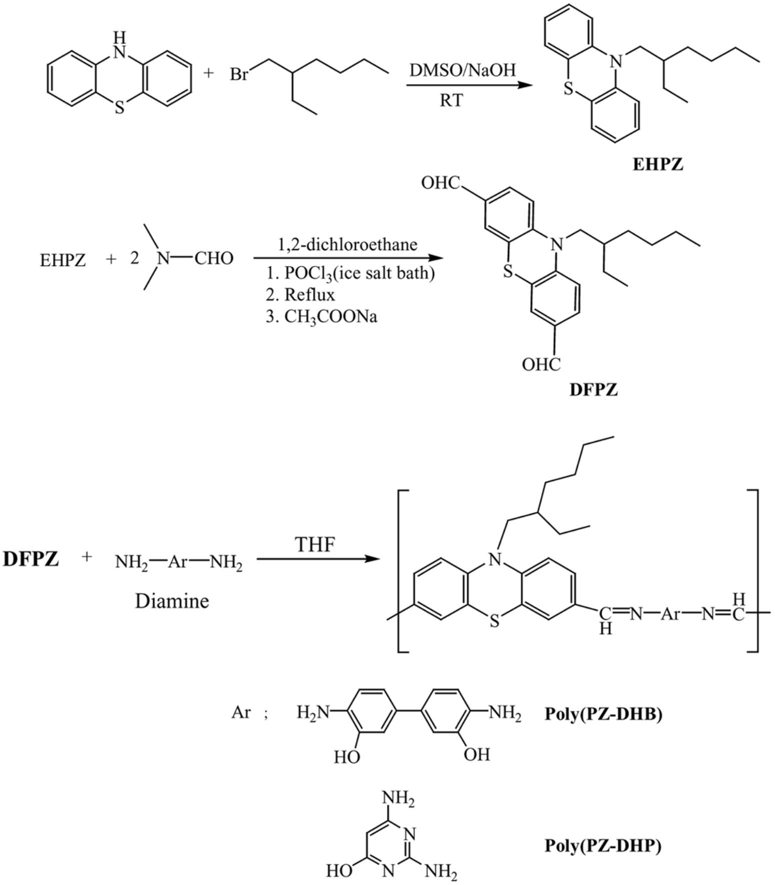

SCHEME 1 Synthetic procedure and structures of poly(PZ-DHP) and poly(PZ-DHB).

2.1. *Synthesis of poly(PZ-DHB)*

2.1.1. N-(2-Ethylhexyl)phenothiazine(EHPZ). First dimethylsulfoxide (DMSO) (60 mL) and sodium hydroxide (7.2 g, 0.18 mol) were charged in a three-necked flask and stirred for 30 min while flushing with nitrogen gas. Phenothiazine (6 g, 0.03 mol) and 2-ethylhexylbromide (5.8 g, 0.03 mol) were added to this suspension solution and the mixture was reacted for 24 hr at room temperature [12,13]. After reaction DMSO was removed by using rotary evaporator. Then acetone was added into the reaction mixture and the resulting insoluble sodium hydroxide was removed by filteration. The product solution

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was purified by extraction with the water and dichloromethane mixture solvent. The crude product obtained form the dichloromethane part was purified by column chromatography by using and *n*-hexane:ethyl acetate (9:1 v/v) as eluent, and then dried to give a transparent liquid product, N-(2-ethylhexyl)phenothiazine(EHPZ).

Yield: 73%, ^{1}H-NMR($CDCl_3$), δ: 6.87, 7.12 (m, 8H, Ar-H), 3.70 (m, 2H, $>N-CH_2-$), 1.93 (t, 1H, $>CH-$), 1.22–1.44 (m, 9H, $-CH_2-$), 0.83–0.87 (m, 6H, $-CH_3$), IR(KBr), cm^{-1}: 3064 (Ar CH), 2926 (aliphatic CH), 2857 ($>N-CH_2-$), 1593, 1485 (Ar C=C).

2.1.2. N-(2-Ethylhexyl)-3,6-diformylphenothiazine(DFPZ). Introduction of aldehyde groups to the EPHZ intermediate was achieved by using Vilsmeier-Haack reaction [10,11]. First EHPZ (12.36 g, 0.04 mol) was dissolved in 1,2-dichlororoethane (30 mL) solvent followed by addition of dimethylformamide (58.5 g, 0.8 mol) reagent. After the solution was cooled to 0°C phosphorus oxychloride (122.7 g, 0.8 mol) was added dropwise for 30 min, and then the mixture was stirred for 72 hr at the refluxing temperature. The mixture was then cooled to 25°C and sodium acetate aqueous solution (33 wt%, 27.34 g) was added. After removal of solvents by evaporation, acetone was added and the insoluble salt was removed by filteration. The product solution was purified by extraction with water and dichloromethane mixture solvent. The crude product obtained from the dichloromethane part was purified by column chromatography using *n*-hexane:ethyl acetate (3:1 v/v) as eluent, and then dried to give the solid product, N-(2-ethylhexyl)-3,6-diformylphenothiazine (DFPZ).

Yield: 40%, ^{1}H-NMR($CDCl_3$), δ: 9.73 (m, 2 H, aldehydic hydrogen), 6.88, 7.56 (m, 6 H, Ar H), 3.73 (t, 2 H, $>N-CH_2-$), 1.17–1.85 (m, 9 H, $-CH_2-$, $>CH-$), 0.76–0.82 (m, 6 H, $-CH_3$), IR(KBr), cm^{-1}: 3048 (Ar CH; weak signal), 2927 (aliphatic CH), 2857 ($>N-CH_2-$), 2724 (aldehydic hydrogen), 1688 ($>C=O$), 1600, 1465 (Ar C=C).

2.1.3. Synthesis of poly(PZ-DHB). The DFPZ (3.67 g, 0.01 mol) monomer synthesized above and another monomer 3,3′-dihydroxybenzidine (2.16 g, 0.01 mol) were dissolved in THF (60 mL) solvent and the mixture solution was refluxed for 24 hr to give a heterocyclic EL polymer with azomethine units, poly(PZ-DHB). After polymerization the product polymer was precipitated with *n*-hexane as non-solvent. The polymer was further purified by reprecipitation and then dried in the vacuum oven for 24 hr at 30°C to give purified product, poly(PZ-DHB).

Yield: 68%, IR(KBr), cm^{-1}: 3027 (Ar CH), 2924 (aliphatic CH), 2857 ($>N-CH_2-$), 1620, 1467 ($-N=C-$), 1580, 1467 (Ar C=C).

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2.2. *Synthesis of Poly(PZ-DHP)*

The DFPZ (3.67 g, 0.01 mol) monomer and 2,4-diamino-6-hydroxypyrimidine (1.26 g, 0.01 mol) was dissolved in DMSO (60 mL) solvent and the mixture solution was refluxed for 24 hr to give another EL polymer, poly(PZ-DHP). The product polymer was purified by reprecipitation using *n*-hexane as non-solvent and then dried under vacuum.

Yield: 78%, IR(KBr), cm^{-1}: 3018 (Ar CH), 2925 (aliphatic CH), 2856 ($>N-CH_2-$), 1625, 1481 (–N=C–), 1579, 1481 (Ar C=C).

2.3. *Synthesis of Photosensitive EL Polymer, poly(PZ-DHB)-PS*

A photosensitive EL polymer, poly(PZ-DHB)-PS was synthesized by introduction of photocrosslinkable units in the side chain as shown in Scheme 2.

2.3.1. Synthesis of hydroxystyryl-2-pyridine (2-HSP). 2-Picoline (12 g, 0.13 mol), *p*-hydroxybenzaldehyde (20.9 g, 0.17 mol) and acetic anhydride (26.3 g, 0.26 mol) were refluxed for 24 hr in a three neck flask equipped a cooling condenser under nitrogen atmosphere. After reaction the intermediate product (2-ASP) was precipitated in a ice-water bath and filtered. Recrystallization of 2-ASP in ethanol gave pure product in 62.5% yield. The intermediate (2-ASP) was added to a mixture solution of 0.5 N KOH/EtOH in a three neck flask and then refluxed for 1 hr. The hydrolyzed product was neutralized by adding 4 ml of 1 N HCl solution followed by filtration. Recrystallization in ethanol gave hydroxystyryl-2-pyridine (2-HSP) in 72.3% yield.

2.3.2. Hydroxyhexyloxystyryl-2-pyridine (2-HHSP). The intermediate 2-HSP (6 g, 0.03 mol) was dissolved in 500 ml acetone in a three neck flask and then K_2CO_3 (12.4 g, 0.09 mol) was added and the mixture was refluxed for 1 hr. Into the mixture solution 6-bromo-1-hexanol was slowly added and the whole mixture was refluxed for 24 hr. After reaction KBr byproduct was removed by filteration and the filterate was passed through a silica gel column with acetone as solvent. The product solution was concentrated by using rotary evaporator and then extracted with *n*-hexane below 4°C. The extracted product was recrystallized in the acetone/ethanol (v/v = 1.5) mixture solvent to give 2-HHSP in 67.3% yield.

2.3.3. Synthesis of photosensitive EL polymer, poly(PZ-DHB)-PS. The substrate polymer, poly(PZ-DHB) (1 eq.), the photosensitive compound, 2-HHSP (1.5 eq.) and triphenyl phosphine (PPh_3) (1.5 eq.) were dissolved in THF solvent in a three neck flask and then diethyl azodicarboxylate(DEAD) (1.5 eq.) was added dropwise over

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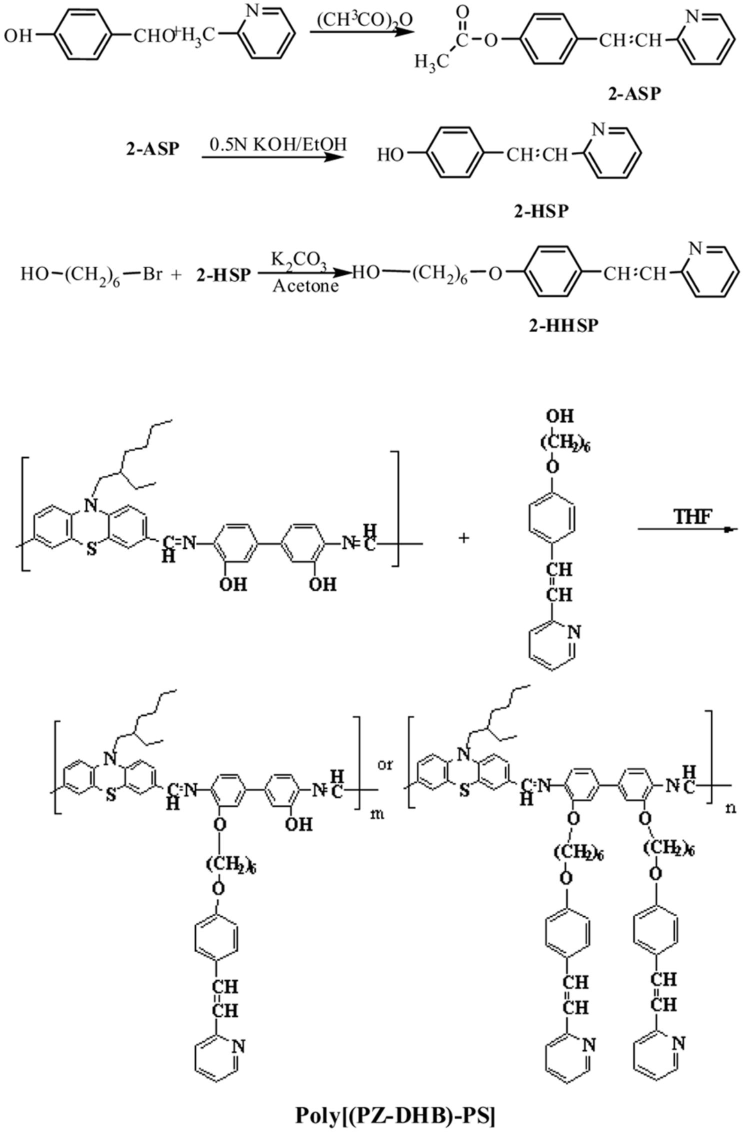

SCHEME 2 Synthetic procedure and the structure of photosensitive polymer, poly[(PZ-DHB)-PS].

1 hr and the whole reaction mixture was reacted for 72 h at room temperature. After reaction THF was removed by rotary evaporator and the product was precipitated with methanol as non-solvent. The

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product polymer was purified by reprecipitation in methanol followed by filteration and drying in vauum oven for 24 hr at 30°C to give a photosensitive EL polymer, poly(PZ-DHB)-PS.

Yield: 62%, IR(KBr), cm^{-1}: 3018 (Ar CH), 2900 (aliphatic CH), 2856 ($>N-CH_2-$), 1635 ($-C=C-$), 1620, 1481 ($-N=C-$), 1579, 1481 (Ar C=C).

3. Measurement

Infrared spectra were obtained with a JASCO FT-IR 620 spectrometer. ^1H-NMR spectra were recorded on a *Bruker* 400 NMR spectrometer with chloroform-*d*($CDCl_3$) as solvent and tetramethylsilane (TMS) as internal reference. UV/visible and fluorescence spectra of synthesized products were obtained with a Simadzu UV-5100 spectrophotometer and JASCO FP-6500 fluorescence spectrometer, respectively. Thermal stability of the compounds was evaluated by using a Seiko SSC5200 H Disk Station (DSC220C) under dry nitrogen at a heating rate of 20°C/min. The average molecular weights (M_w and M_n) of the polymers were measured with a gel permeation chromatograph (Waters, alliance 2000, eluent THF, polystyrene universal calibration) at room temperature.

RESULTS AND DISCUSSION

1. Synthesis and Characterization

In the synthesis of heterocyclic conjugated polymers, poly (phenoxiazine-3,3′-dihydroxybensidine)(PZ-DHB) and poly(phenoxiazine-2,4-diamino-6-hydroxypyrimidine)(PZ-DHP), it was found that the Schiff-base reaction [14,15] was useful to obtain polycondensation product with relatively high molecular weights and yield. As shown in Table 1 the yields of poly(PZ-DHB) and poly(PZ-DHP) were 68% and 78%, respectively. The thermal stability was also high as confirmed by initial thermal decomposition temperatures (T_{id}) of 320°C and 230°C, respectively. The lower thermal decomposition temperature of poly(PZ-DHP) than poly(PZ-DHB) seemed to be due to rather low

TABLE 1 Characterization Data of Poly(PZ-DHB) and Poly(PZ-DHP)

Polymer	Yield (%)	$M_w(\times 10^{-4})$	$M_n(\times 10^{-4})$	T_{id}
Poly(PZ-DHB)	68	1.14	0.77	320
Poly(PZ-DHP)	78	0.96	0.65	230

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molecular weight (M_w) of the polymer. The M_w of poly(PZ-DHB) was determined to be 1.12×10^4 and M_w/M_n value was 1.45, however, M_w of poly(PZ-DHP) could not be measured by GPC due to lack of solubility in common solvents. The poor solubility but higher yield of poly(PZ-DHP) than poly(PZ-DHB) suggested that poly(PZ-DHP) chain was much stiffer than poly(PZ-DHB) which has biphenyl joint unit in the main chain, thus leading to premature precipitation during the polymerization reaction.

The introduction of photocrosslinkable moiety (2-HHSP) to poly(PZ-DHB) was achieved through Mitsunobu reaction by using triphenyl phosphine and diethylazodicarboxylated (DEAD) reagents [16,17]. The photocrosslinkable conjugated polymer, poly[(PZ-DHB)-PS] was obtained in 65% yield and the 2-HHSP photocrosslinkable unit was confirmed by the characteristic C=C bond at 1635 cm^{-1} as shown in Figure 1.

2. Optical and Photocrosslinking Properties

UV-visible and PL spectra of poly(PZ-DHB) and poly(PZ-DHP) in 5×10^{-5} M solution of THF are shown in Figure 2. The UV absorption of poly(PZ-DHB) exhibited two peaks at 300 and 430 nm corresponding

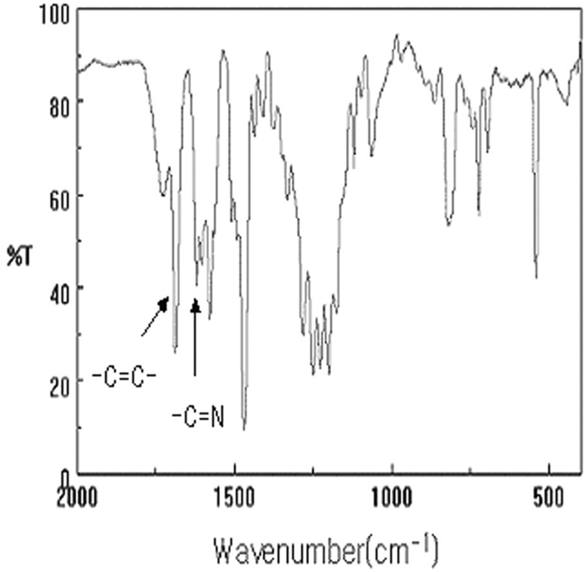

FIGURE 1 FT-IR spectrum of photosensitive polymer, poly[(PZ-DHB)-PS].

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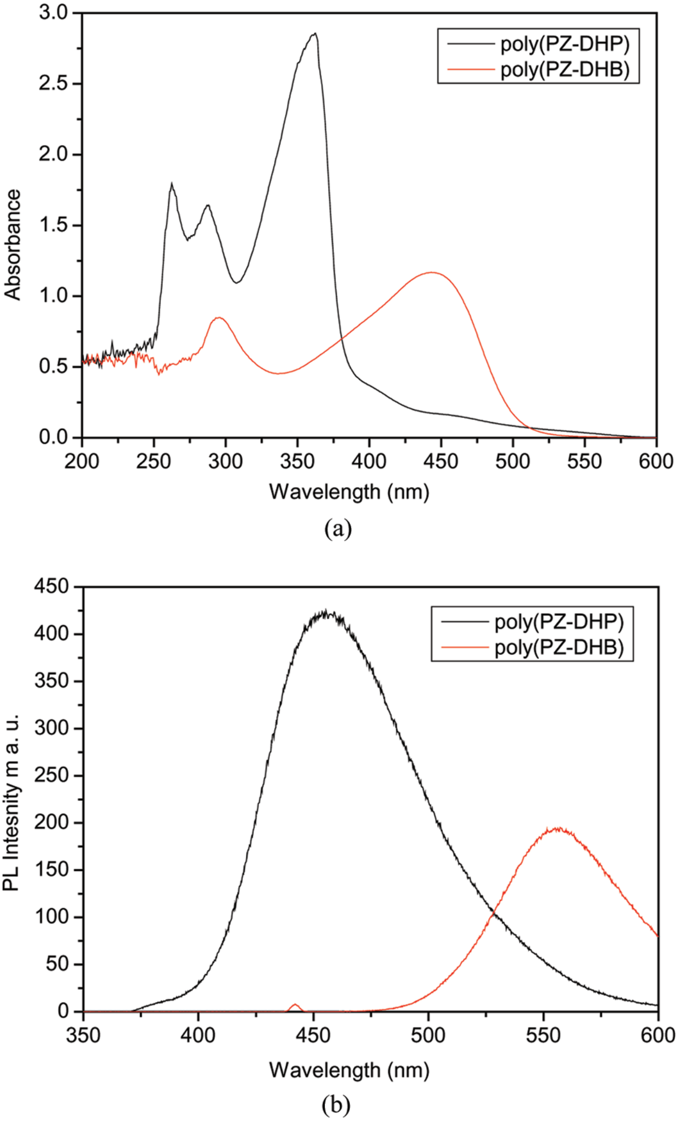

FIGURE 2 UV absorption (a) and fluorescence spectra (b) of poly(PZ-DHP) and poly(PZ-DHB) (λB$_{\text{exB}}$ = 400 nm, c = N5.0 × 10P^{-5} M in THF).

to π-π^* transition of azomethine and azo group, respectively. The UV absorption of azomethine in poly(PZ-DHP) was shifted to longer wavelength (362 nm) compared with that of poly(PZ-DHB) due to the non-bonding electrons of nitrogen atom in the pyrimidine ring. The UV absorption poly[(PZ-DHB)-PS] exhibited similar peaks except a peak at 330 nm due to the strylylpyridyl group.

When three polymer, poly(PZ-DHP), poly(PZ-DHB) and poly[(PZ-DHB)-PS] were excited at the UV maximum absorption ($\lambda_{\mathrm{max,abs}}$), their fluorescence spectra showed the maximum emission ($\lambda_{\mathrm{max,em}}$) at 462, 560 and 555 nm, respectively. The conjugated polymer poly(PZ-DHP) with azomethine unit exhibited strong blue emission, while poly(PZ-DHB) and poly[(PZ-DHB)-PS] emitted greenish yellow color due to increased conjugation compared to poly(PZ-DHP).

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3. Photosensitive Properties of Poly[(PZ-DHB)-PS]

Figure 3 shows the UV adsorption spectra of poly(PZ-DHB)-PS in THF solution (5×10^{-5} M) and in the film state under varying UV exposure dose. The UV spectra both in solution and film state exhibited that UV absorption at 330 nm decreased with increasing UV dose and became constant under 1.3 J/cm^2.

These results would be explained by the [2 + 2] photocycloaddition reaction of styrylpyridyl groups present in the side chains of poly(PE-DHB)-PS [18,19].

The change of normarlized UV absoptions of poly(PZ-DHB)-PS both in solution and film state at 330 nm are shown in Figure 4 according to the UV exposure dose. It was noted from Figure 4 that the UV absorption decreased fast in solution but in film state decreased slowly with the UV exposure dose, which could be explained by the restricted movement of the pendant styrylpyridyl groups in the film state.

The FT-IR spectra of poly(PZ-DHB)-PS films before (a) and after (b) UV exposure are shown in Figure 5. The photo-activity of poly (PZ-DHB)-PS was determined to be 65% by comparing the absorbance of C=N(at 1380 cm^{-1}) and C=C(at 1635 cm^{-1})in film state according to equation(1).

$$\text{Photo-reactivity}(\%) = 100 - [(I_{(C=C)}/I_{(CN)})/(I_{O(C=C)}/I_{O(CN)}) \qquad (1)$$

where, $I_{O(C=C)}$ = Area of C=C peak before UV exposure, $I_{O(CN)}$ = Area of −CN peak before UV exposure, $I_{(C=C)}$ = Area of C=C peak before UV exposure (2.0 J/cm^2), $I_{(CN)}$ = Area of −CN peak before UV exposure (2.0 J/cm^2).

The patterning property of the conjugated polymer, poly(PZ-DHB)-PS, was checked by the following photolithographic process. The poly(PZ-DHB)-PS solution in THF (2 wt%) was spin-coated on a

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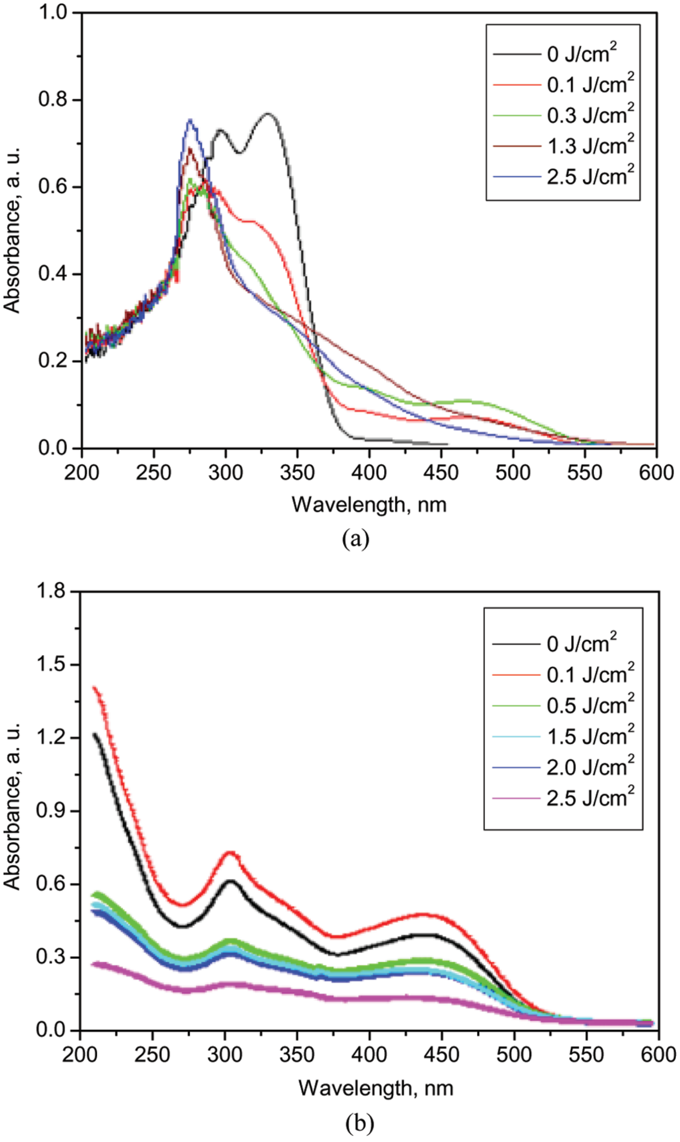

FIGURE 3 UV spectra of poly(PZ-DHB)-PS in THF solution (a) and in film state (b) when exposed to unpolarized UV light at varying dose.

cleaned ITO-glass at a series of speed, 500 rpm for 5 sec and 2500 rpm for 25 sec. The cast film was dried on an IR oven at 110°C for 1 hr. The resulting thin film was exposured to UV light to the dose of

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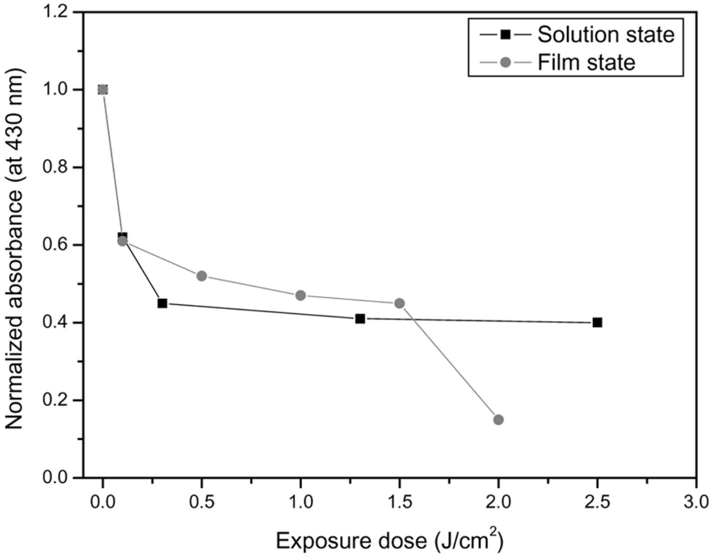

FIGURE 4 The change of normalized absorbance of poly(PZ-DHB)-PS in THF solution and in film state according to the UV dose.

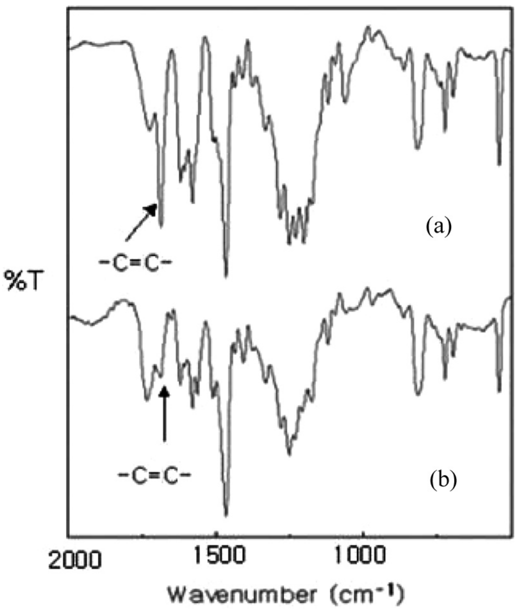

FIGURE 5 FT-IR spectra of poly(PZ-DHB)-PS films before (a) and after (b) irradiation of unpolarized UV light.

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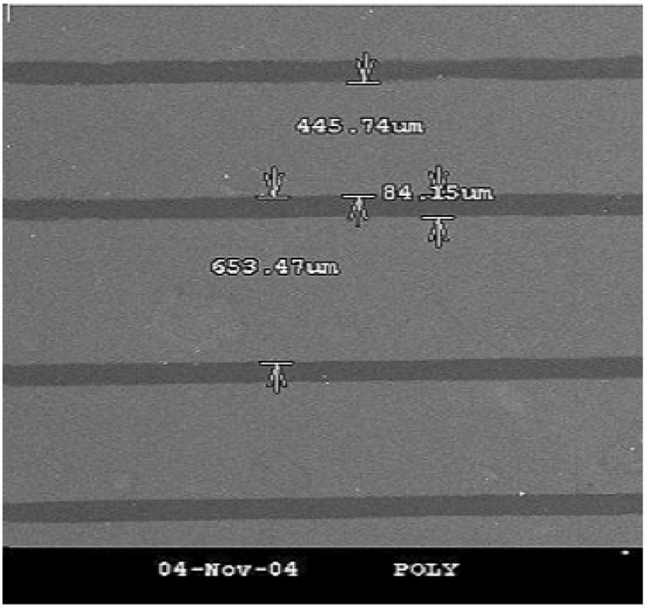

FIGURE 6 The SEM image of poly(PZ-DHB)-PS thin film after photolithographic process.

2000 mJ/cm^2 through a photomask in a contact mode. The poly (PZ-DHB)-PS film gave a fine pattern with line width (80 μm), space (445 μm) and thickness (2 μm) as shown in Figure 6.

The photosensitivity of conjugated polymer is a desirable property for the fabrication of polymer light emitting diode (PLED). The performance of poly(PZ-DHB)-PS in the PLED devices will be reported else where.

CONCLUSION

Conjugated polymers containing phenothiazine moiety and azomethine linkage in the main chain were synthesized by Schiff-base polycondensation. The photosensitive version of the polymer, poly (PZ-DHB)-PS, was also synthesized by introduction of styrylpyridyl group in the side chins.

The photosensitive polymer exhibited good solubility in common organic solvents and strong PL emission at 555 nm. The photosensitive polymer was also found to have good patterning property in the standard photolithographic process.

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