

# Synthesis, characterization and fluorescence properties of azomethine polymer containing quinoline unit

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Received: 14 March 2017 / Revised: 8 July 2017 / Accepted: 11 July 2017 /

Published online: 15 July 2017

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**Abstract** In this study, synthesis, characterization and photophysical properties of the quinoline and azomethine derived polymers were described with ether linkages. Synthesis procedure was conducted in three steps; the first step, dialdehyde monomer, the second step poly(azomethine) (PA), and the third step poly(azomethine-quinoline) (PAQ) polymers were obtained with elimination and condensation reactions. The structures of synthesized compounds were confirmed with FT-IR, UV-Vis and <sup>1</sup>H-<sup>13</sup>C-NMR spectra. Electrochemical and photophysical behaviors were interpreted with cyclic voltammetry and photoluminescence data. In addition, thermal properties of compounds were obtained by TG and DSC techniques. Surface images of polymers were photographed with scanning electron microscope (SEM). Electrochemical band gaps ( $E'_{g1}$ ) of PA and PAQ were calculated as 2.19, 1.83 eV, respectively. The starting degradation temperatures of PA and PAQ were found as 126 and 184 °C, respectively.

**Keywords** Electrochemical properties · Fluorescence properties · Poly(azomethine-quinoline) · SEM · Thermal degradation

## Introduction

N-Hetero or cyclic inclusive polymers having n-type electronic conducting features are particular interest for us today. Quinoline inclusive materials have been substantially developed for practices as electronic transport equipages in light-emitting diodes by virtue of their electronic and optical properties [1–6].

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Poly(quinoline)s have possessed n-type electronic conducting features along with high thermal, mechanical and oxidative features. Certain proceedings on quinoline inclusive polymers with confined practice have been executed on biquinoline inclusive polymers [7–18]. Recently, conjugated quinoline derivatives molecules have produced significant relevance as blue-emitting requisites [19–21]. Conjugating poly(quinoline)s have revealed significant electronic transport and photoluminescent features in organic light-emitting diodes (OLEDs) [22, 23]. Poly(quinoline)s are novel as very hopeful blue-emitting requisites by virtue of having matchless assembly of high thermal character, simple workability and high photoluminescence quantum yields [24]. Various quinoline-containing products have also been worked in molecularly doped polymer LEDs [25, 26].

Poly(azomethine)s, poly(imine)s or poly(Schiff bases) are another interesting sort of conjugated polymers having nitrogen atoms in imine (N=CH) group of polymer chain. These polymers are a class of high performance molecules as they present a number of interesting and unique properties such as preeminent thermal, film-making ability, mechanic and physical properties in several domains, e.g., electronic, photonics and industrial materials [27].

In this paper, we have aimed to synthesize polymers that contain azomethine and quinoline units in main chain and side chain, respectively. These processes were realized by elimination reactions on three steps. Then, the structures of these compounds were confirmed with FT-IR, UV–Vis and <sup>1</sup>H-NMR measurements. In addition, thermal, electrochemical and photophysical behaviors of obtained compounds were evaluated with TG-DTG, DSC, CV and PL techniques.

## Experimental

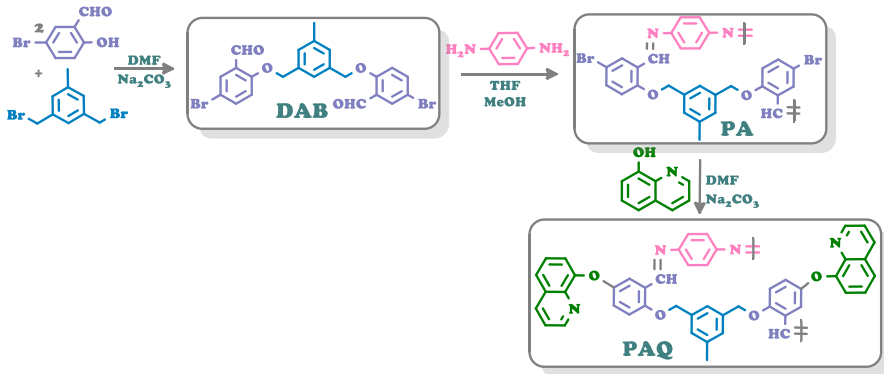
### Chemicals

5-Bromo-salicylaldehyde, 8-hydroxyquinoline and *p*-phenylenediamine were supplied by Sigma Aldrich and 3,5-bis(bromomethyl)toluene were supplied by Alfa Aesar. Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), dimethylformamide (DMF), methanol (MeOH), ethanol (EtOH), chloroform, ethyl acetate, hexane, dimethyl acetamide (DMA), dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), and acetonitrile were purchased from Merck Chem. Co. (Germany).

### Synthesis of poly(azomethine) and poly(azomethine-quinoline)

The synthesis procedure of the first product is dialdehyde bromide (DAB), the second product is poly(azomethine) (PA) and the third product is poly(azomethine-quinoline) (PAQ); these are demonstrated in Scheme 1.

In the first step, 5-bromo-salicylaldehyde (0.04 mol) and dry DMF (25 mL) were charged into three different 250 mL flasks. An amount of 0.02 mol of Na<sub>2</sub>CO<sub>3</sub>, dissolved in DMF (5 mL), was added to this mixture and heated (60 °C, 1 h) with stirring under argon atmosphere. An amount of 0.02 mol 3,5-bis(bromomethyl) toluene was dissolved in DMF (30 mL), and then, it was added to this mixture and



**Scheme 1** Synthesis of poly(azomethine) and poly(azomethine-quinoline)

refluxed (160 °C, 8 h). This reaction solution was sloped into 100 mL of iced distilled water and the settled pure products were gathered. The yield of obtained aromatic dialdehyde bromide (DAB) was found as 75%.

In the second step, obtained DAB (0.02 mol) was dissolved in THF (25 mL). An amount of 0.02 mol of *p*-phenylenediamine, dissolved in methanol (5 mL), was added to this mixture and heated (60 °C, 24 h) slowly. This mixture was filtered, recrystallized from acetonitrile and dried in vacuum desiccators [28], and the yield of poly (azomethine) (PA) was found as 65%.

In the third step, obtained PA (0.02 mol) was dissolved in DMF (10 mL). An amount of 0.02 mol of 8-hydroxyquinoline and 0.02 mol  $\text{Na}_2\text{CO}_3$  dissolved in DMF (5 mL) were added to this mixture and heated (160 °C, 36 h) with stirring under argon gas. This reaction solution was added into 100 mL of iced distilled water and the settled pure products were gathered. The yield of obtained poly(azomethine-quinoline) (PAQ) was found as 50%.

### Characterization techniques

FT-IR spectra were measured by PerkinElmer Spectrum One FT-IR system and recorded using universal ATR sampling accessory in the mid-IR region between 4000 to 650  $\text{cm}^{-1}$ . UV-Vis spectra were recorded within the wavelengths of 200–800  $\text{cm}^{-1}$  by Analytik Jena Specord 210 Plus. NMR spectra of the samples were monitored in a Bruker Avance DPX-600 MHz instrument using  $\text{DMSO-d}_6$  as solvent at 298 K. The number average molecular weight ( $M_n$ ), weight average molecular weight ( $M_w$ ) and polydispersity index (PDI) were determined by Gel Permeation Chromatography–Light Scattering (GPC–LS) device of Malvern Viscotek GPC Dual 270 max. For GPC investigations, an SGX (100 Å and 7 nm diameter loading material) 8 mm i.d.  $\times$  300 mm dual columns was used. Addition of 1 g  $\text{L}^{-1}$  of lithium bromide in DMF (1 mL  $\text{min}^{-1}$ ) was used as solvent. Light scattering detector (LS), a refractive index detector (RID) and polystyrene standards were used to analyze the polymers at oven 55 °C [29]. The cyclic voltammetry (CV) measurements of compounds were carried out by a device CHI 660 C electrochemical analyzer (CH instruments, Texas, USA) at

various potential scan rates as given in the literature [29]. The surface morphology of polymers was recorded by a Jeol JSM-7100F Schottky field emission scanning electron microscope. Sputter coating process was used to create a thin gold/palladium film onto the polymer particles. Photoluminescence (PL) measurements were carried out by Shimadzu RF-5301PC spectrofluorophotometer. DSC thermogram was recorded by PerkinElmer Pyris Sapphire DSC in the range of 25–450 °C in an aluminum pan (in N<sub>2</sub>, 10 °C min<sup>-1</sup>). Al<sub>2</sub>O<sub>3</sub> is used as standard compound in DSC measurement. The thermogravimetric measurements were carried out with TG-DTA PerkinElmer Diamond system apparatus under various heating rates from ambient temperatures up to 1000 °C. The thermograms were recorded using a platinum crucible as sample container in dynamic nitrogen atmosphere at a flow rate of 100 mL min<sup>-1</sup>. In all thermal measurements, the samples were used as 10 mg of weight.

## Results and discussion

### Solubility and spectral properties of compounds

The solubility tests were performed by solution/compound systems (1 mg 1 mL<sup>-1</sup>) at room temperature. As a result of performed solubility tests, the PAQ was found to be sparingly soluble in all selected solvents. While the PA was found to be insoluble in alcohols, chloroform and nonpolar solvent such as hexane, it was soluble in high polar solvents. Also, the DAB was found to be soluble in high polar solvents such as DMSO, DMA, DMF and THF (Table 1). These soluble features make these compounds more feasible and applicative for various potential areas.

FT-IR spectra of monomer and polymers are given in Fig. 1. Spectra of DAB have specifically three peaks at 1681 cm<sup>-1</sup> related to aldehyde, 1269 cm<sup>-1</sup> related to –C–O–C– and 674 cm<sup>-1</sup> related to –C–Br stretch vibrations. The FT-IR spectra of PA have specifically peaks at 1610 cm<sup>-1</sup> related to –C=N– (imine), 1264 cm<sup>-1</sup> related to –C–O–C– and 674 cm<sup>-1</sup> related to –C–Br stretch vibrations. Moreover, spectra of PAQ have specifically peaks at 1618 cm<sup>-1</sup> related to –C=N– (imine) and 1264 cm<sup>-1</sup> related to –C–O–C– stretch vibrations. It is seen that in Fig. 1, the peaks were wider at the spectra of PAQ. All these results are confirmed structures of monomer and polymers.

**Table 1** Solubility results of synthesized compounds

	DAB	PA	PAQ
EtOH	–	–	+
MeOH	–	–	+
Chloroform	–	+	+
THF	+	+	+
DMF	+	+	+
Hexane	–	–	+
DMA	+	+	+
DMSO	+	+	+

+ soluble, – insoluble

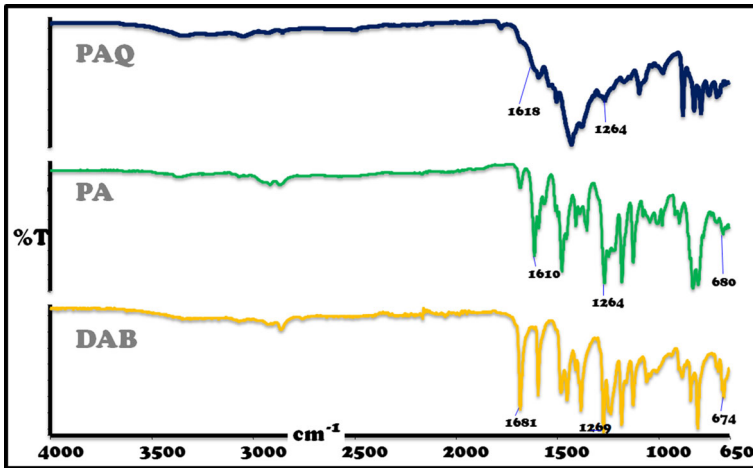


Fig. 1 FT-IR spectra of DAB, PA and PAQ

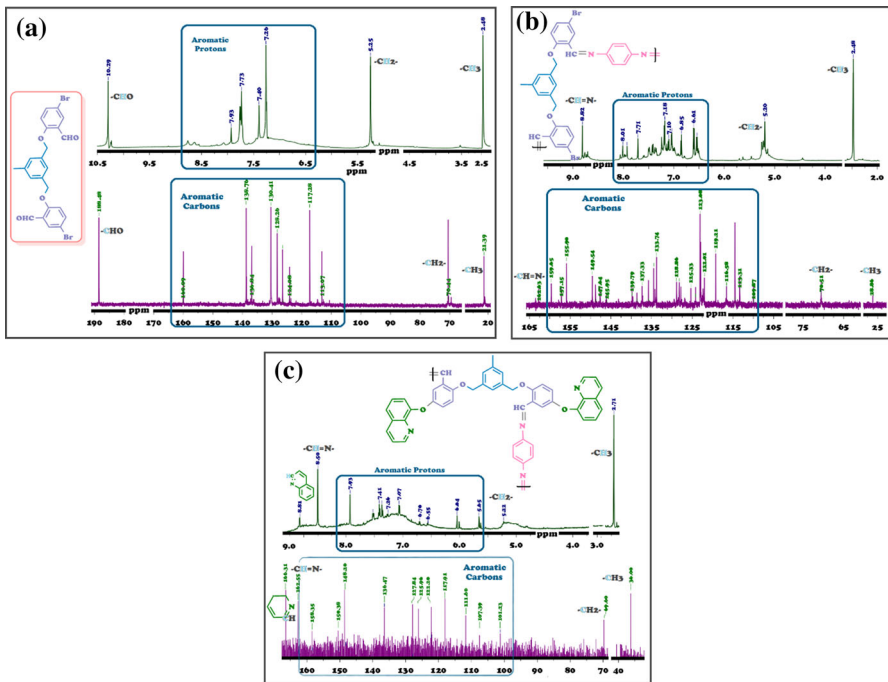


Fig. 2 <sup>1</sup>H-<sup>13</sup>C-NMR spectra of DAB (a), PA (b) and PAQ (c)

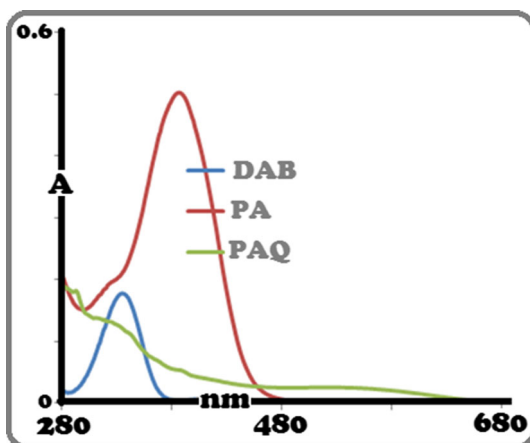
The <sup>1</sup>H-<sup>13</sup>C-NMR spectra of DAB, PA and PAQ are given in Fig. 2. The aldehyde, aromatic, and aliphatic (methylene and methyl) proton signals of DAB were observed at 10.29, 7.26–7.93 and 5.25 and 2.48 ppm, respectively, in the <sup>1</sup>H-NMR spectra. Also, imine (–CH=N), aromatic and aliphatic (–CH<sub>2</sub> and –CH<sub>3</sub>)

proton signals of PA were observed at 8.82, 6.55–8.01 and 5.20 and 2.48 ppm, respectively, in the  $^1\text{H}$ -NMR spectra. The imine ( $-\text{CH}=\text{N}$ ), imine of quinoline ring, aromatic and aliphatic ( $-\text{CH}_2$  and  $-\text{CH}_3$ ) proton signals of PAQ were observed at 8.50, 8.81, 5.65–7.93 and 5.22 and 2.71 ppm, respectively, in the  $^1\text{H}$ -NMR spectra. The aldehyde ( $-\text{CHO}$ ), aromatic, and aliphatic (methylene and methyl) carbon atom signals of DAB were observed at 188.48, 119.07–160.09 and 70.44 and 21.39 ppm, respectively, in the  $^{13}\text{C}$ -NMR spectra. The imine ( $-\text{CH}=\text{N}$ ), aromatic and aliphatic ( $-\text{CH}_2$  and  $-\text{CH}_3$ ) carbon atom signals of PA were observed at 162.63, 109.07–159.65 and 70.51 and 28.86 ppm, respectively, in the  $^{13}\text{C}$ -NMR spectra. The imine ( $-\text{CH}=\text{N}$ ), imine of quinoline ring, aromatic and aliphatic ( $-\text{CH}_2$  and  $-\text{CH}_3$ ) carbon atom signals of PAQ were observed at 162.55, 166.31, 101.23–158.35 and 69.60 and 36.00 ppm, respectively, in the  $^{13}\text{C}$ -NMR spectra.  $^1\text{H}$ - $^{13}\text{C}$ -NMR analyses results were confirmed to be formation of DAB, PA and PAQ compounds. According to GPC measurement,  $M_w$  and PDI values of PA and PAQ were determined as 6800 and 12,500 Da and 1.40 and 1.45, respectively. According to these values, repeated unit numbers of PA and PAQ were found as 11 and 17, respectively.

To elucidate the electronic states of the product, UV–Vis absorption measurements were performed. Figure 3 shows UV–Vis spectra of purely synthesized DAB, PA and PAQ, and the findings and data are also situated in Table 2. The optical band gap ( $E_g$ ) values of DAB, PA and PAQ were calculated using  $E_g = 1242/\lambda_{\text{on}}$  equation, as given in the literature [30], and found as 2.64, 2.67 and 1.94 eV, respectively. Due to extended conjugated structure, PAQ exhibits lower band gap than that of PA as expected. The observed optic band gap value of PAQ is sufficiently low to make this polymer highly promising for photovoltaic applications [30].

DAB showed an absorption band in the UV–Vis spectra. The absorption band is sighted between 290 and 375 nm owing to  $\pi \rightarrow \pi^*$  transition of benzene conjugation. PA showed two absorption bands in the UV–Vis spectra. The first absorption band is sighted between 295 and 305 nm owing to  $\pi \rightarrow \pi^*$  transition of

**Fig. 3** Absorption spectra of DAB, PA and PAQ



**Table 2** Optical band gap,  $\lambda_{\max}$ ,  $\lambda_{\text{onset}}$  values of the compounds

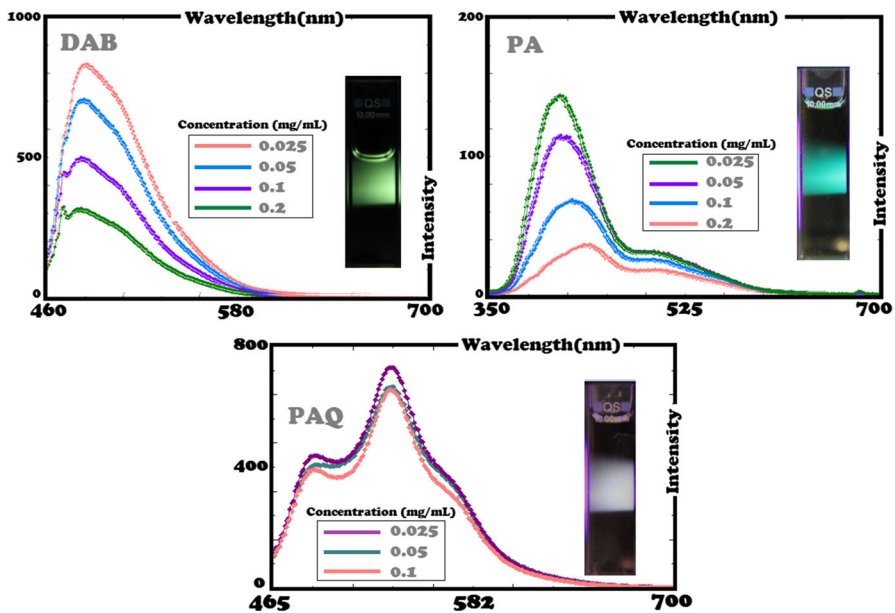
Compounds	$\lambda_{\max}$ (nm)	$\lambda_{\text{onset}}$ (nm)	$E_g$ (eV) <sup>a</sup>
DAB	305, 440	470	2.64
PA	300, 432	465	2.67
PAQ	290, 412, 550	640	1.94

<sup>a</sup> Optical band gap

benzene conjugations. Second absorption band is sighted in the range 305–465 nm owing to  $\pi \rightarrow \pi^*$  transition of imine conjugations, respectively. Moreover, PAQ showed two absorption bands in the UV–Vis spectra. The first absorption band is sighted between 300 and 475 nm owing to  $\pi \rightarrow \pi^*$  transition of benzene conjugations. The second absorption band is sighted in the range 475–640 nm owing to  $\pi \rightarrow \pi^*$  transition of imine conjugations, respectively [31].

### Fluorescence properties

Photoluminescence (PL) spectroscopy and obtained data were used to measure light reactions of the polymers (Fig. 4). The PL spectra of DAB, PA and PAQ were obtained in DMF solutions. Solution concentrations of the compounds and slit width of the spectrofluorophotometer were modulated between 0.1 and 0.05 mg L<sup>-1</sup> and 3 nm for all measurements, respectively. PL spectra of obtained compounds have emission maxima for DAB, PA and PAQ at 475 nm, 445 nm, and 435 nm,

**Fig. 4** PL spectra of synthesized DAB, PA and PAQ

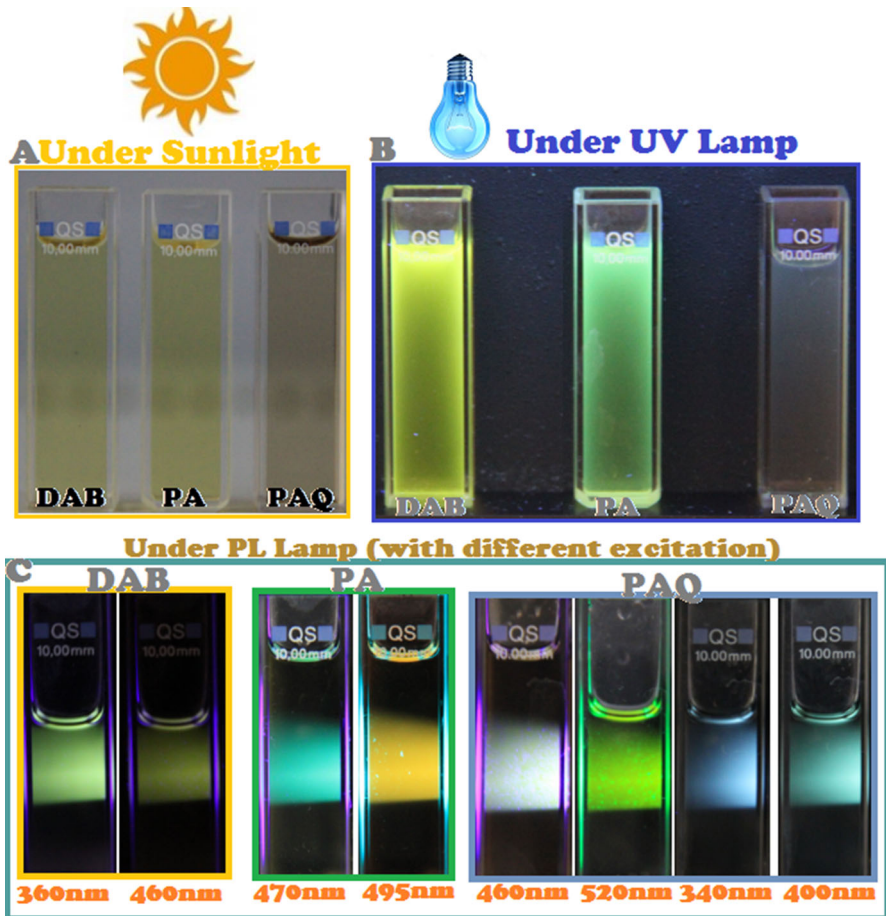
**Table 3** Photoluminescence values of the DAB, PA and PAQ

Products	Concentration (mg mL <sup>-1</sup> )	$\lambda_{\text{Ex}}^{\text{a}}$	$\lambda_{\text{max}}^{\text{b}}$ (Em)	$I_{\text{Em}}^{\text{c}}$
DAB	0.1	450	475	875
PA	0.05	340	445	147
PAQ	0.05	330	435	191

<sup>a</sup> Excitation wavelength for emission

<sup>b</sup> Maximum emission wavelength

<sup>c</sup> Maximum emission intensity



**Fig. 5** Appearance of DAB, PA and PAQ in DMF solutions under sunlight (a), UV lamp (b) and PL lamp (c)

respectively [32] (Table 3). In addition, some photographs of PA and PAQ were taken under sunlight and UV lamp (366 nm) in Fig. 5. It is seen in Fig. 5a that DAB and PA were having different yellow color tones. Also, DMF solution of PAQ was



brown. It is seen in Fig. 5b that though DAB was yellow emission, PA and PAQ were green and gray emissions, respectively, in DMF solution under UV lamp (at 366 nm). It is shown in Fig. 5c that different color emissions were observed in DMF solutions with different excitations. For example, DAB was demonstrated as dichromic property when emitted at 360 nm (green) and 460 nm (yellow). The PA was demonstrated as dichromic property when emitted at 470 nm (turquoise) and 495 nm (bright yellow). The PAQ was demonstrated as multichromic property when emitted at 460 nm (white), 520 nm (green), 340 nm (blue) and 400 nm (turquoise). The PAQ had white emission between 460 and 650 nm (in Figs. 4, 5). Therefore, it can be used as white luminescent material with such rare property.

### Electrochemical properties

Electrochemical characteristics of DAB, PA and PAQ are calculated by cyclic voltammetry (CV) with three electrodes in an electrochemical cell. Highest occupied molecular orbital (HOMO), lowest unoccupied molecular orbital (LUMO) and electrochemical band gap ( $E'_g$ ) data of compounds were found owing to solving electronic structures with reduction and oxidation potentials. Figure 6 demonstrates cyclic voltammograms of compounds. The HOMO, LUMO and  $E'_g$  data of DAB, PA and PAQ were situated as in the literature [29].

The band gaps for synthesized compounds were calculated electrochemically with the equation given below,

$$E_{\text{HOMO}} = -(4.39 + E_{\text{ox}})$$

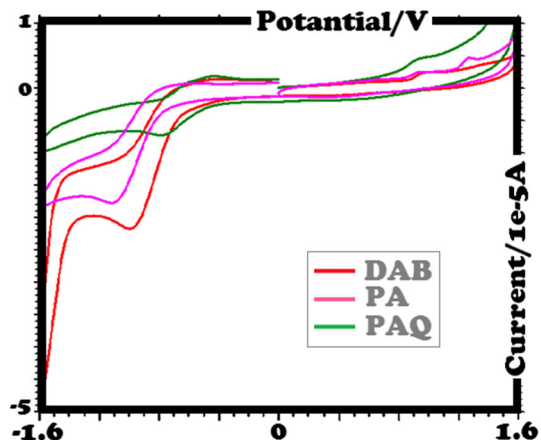
$$E_{\text{LUMO}} = -(4.39 + E_{\text{red}})$$

$$E'_g = E_{\text{LUMO}} - E_{\text{HOMO}}$$

$E_{\text{ox}}$  and  $E_{\text{red}}$  are related to the oxidation and reduction peak potentials. Figure 6 shows CV measurements of DAB, PA and PAQ [33] and they are given in Table 4.

First onset oxidation potentials ( $E_{\text{ox},1}$ ) of DAB, PA and PAQ are 0.97, 0.97 and 0.93 V, respectively, and second onset oxidation potentials ( $E_{\text{ox},2}$ ) are 1.35, 1.25 and

**Fig. 6** Cyclic voltammograms of DAB, PA and PAQ (scan rate:  $-1.6$  to  $1.6$  V  $\text{s}^{-1}$ )



**Table 4** Electrochemical data of DAB, PA and PAQ

Compounds	$E_{ox,1}$ (V)	$E_{ox,2}$ (V)	HOMO <sub>1</sub> (eV) <sup>a</sup>	HOMO <sub>2</sub> (eV) <sup>a</sup>	$E_{red.}$ (V)	LUMO (eV) <sup>b</sup>	$E'_{g1}$ (eV) <sup>c</sup>	$E'_{g2}$ (eV) <sup>c</sup>
DAB	0.97	1.35	−5.38	−5.74	−1.24	−3.15	2.24	2.59
PA	0.94	1.25	−5.33	−5.64	−1.25	−3.14	2.19	2.50
PAQ	0.93	1.49	−5.33	−5.88	0.90	−3.49	1.83	2.39

<sup>a</sup> Highest occupied molecular orbital

<sup>b</sup> Lowest unoccupied molecular orbital

<sup>c</sup> Electrochemical band gap

1.49 V, respectively. HOMO<sub>1</sub> and HOMO<sub>2</sub> energy levels of DAB, PA and PAQ were determined as −5.38, −5.33, −5.33 eV and −5.74, −5.64, −5.88 eV, respectively. Likewise, the onset reduction potentials ( $E_{red.}$ ) of DAB, PA and PAQ are −1.24, −1.25, 0.90 V, respectively, and LUMO energy levels of these compounds were calculated as −3.15, −3.14, −3.49 eV, respectively. Electrochemical band gaps ( $E'_{g1}$  and  $E'_{g2}$ ) of DAB, PA and PAQ were also calculated as 2.24, 2.19, 1.83 eV and 2.59, 2.50, 2.39, respectively. The  $E'_{g1}$  and  $E'_{g2}$  values of PAQ were found as lower than other compounds.

## Thermal properties

Thermal degradation and glass transition temperature ( $T_g$ ) of synthesized compounds were found with TG–DTG–DTA and DSC techniques. The obtained curves from these techniques were demonstrated in Fig. 7. Thermal analysis data of all compounds are given in Table 5. In reference to TG–DTG curves, onset temperatures of synthesized compounds DAB, PA and PAQ were determined as 102, 126 and 184 °C, respectively. The 50% weight loss temperature values of DAB, PA and PAQ were found as 297, 283 and 806 °C, respectively. % chars of DAB, PA and PAQ compounds were found as 2.05, 17.40 and 35.55%, respectively, at 1000 °C. These results are shown that PA and PAQ polymers to be resistant to high temperatures. Similar results are given in the literature [34]. The water or organic solvent loss was observed as 3% in the thermogram of PAQ. All compounds were degraded in three steps. The degradation steps and their values are given in Table 5. According to these results, thermal resistant of PAQ was the highest. According to DSC curves, glass transition temperature ( $T_g$ ) and  $\Delta C_p$  values of PA and PAQ were found as 110 and 145 °C, and 0.075 and 0.173 J g<sup>−1</sup> K<sup>−1</sup>, respectively.

## Surface properties

Surface morphology of obtained PA and PAQ polymers was imaged and investigated by SEM techniques, as shown in Fig. 8. The first photograph belongs to PA polymer and second photograph belongs to PAQ polymer. According to SEM

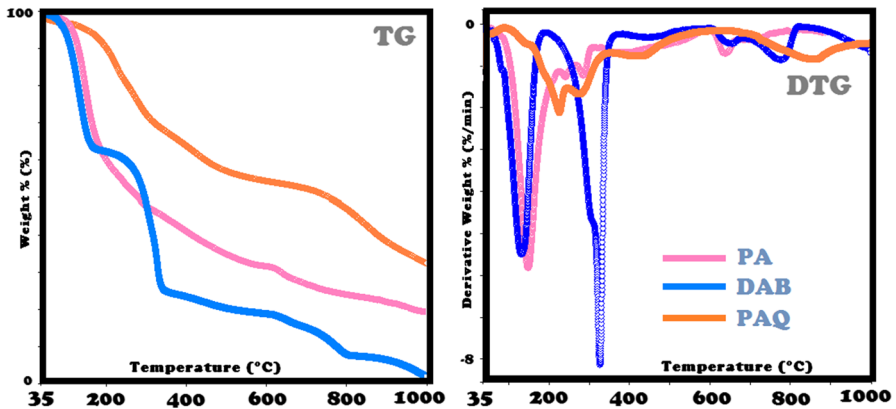
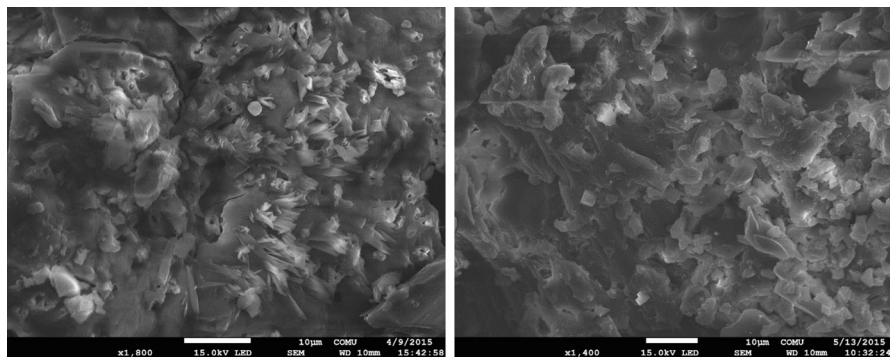


Fig. 7 TG and DTG curves of the synthesized DAB, PA and PAQ

**Table 5** Thermal degradation values of DAB, PA and PAQ

Compounds	DAB	PA	PAQ
The first step			
$T_{on}^a$	102	126	184
$T_{max}^b$	130	148	225, 273
$T_{end}^c$	195	225	355
$\%^d$	37.57	42.49	29.80
The second step			
$T_{star}^e$	195	225	355
$T_{max}$	326	285	424
$T_{end}$	390	325	620
$\%$	38.63	10.40	13.25
The third step			
$T_{star}$	390	325	620
$T_{max}$	776	638	856
$T_{end}$	1000	1000	1000
$\%$	21.75	29.71	21.40
$T_{20}^f$	128	147	272
$T_{50}^g$	297	283	806
Char ( $\%^h$ )	2.05	17.40	35.55

photographs of polymers, azomethine and ether groups containing PA is rigid and uneven; azomethine, quinoline and ether groups containing PAQ is rough and softer than PA but both images of polymers are inhomogeneous [35].



**Fig. 8** FE-SEM images of PA and PAQ polymers, respectively

## Conclusions

Poly(azomethine) and poly(azomethine-quinoline) were synthesized purely from dialdehyde monomer with elimination reactions on three steps. The chemical structures of synthesized these compounds were successfully characterized with by  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$ , FT-IR and UV-Vis spectroscopies. The thermal data obtained from TG-DTA and DSC measurements were shown to be having high char of PA and PAQ polymers such as 17.40 and 35.55%, respectively at 1000 °C. These thermal-resistant polymers have shown low onset and glass transition temperature. Also, in the first step, synthesized DAB monomer has high fluorescence character. According to cyclic voltammetric and UV-Vis spectroscopic results, quinoline-containing PAQ has high optic and electrochemical band gaps such as 1.94 and 1.83 eV, respectively, compared to others. All results demonstrated that PAQ-containing quinoline can be a good candidate for electronic and thermal-resistant materials such as thermally stable light-emitting materials, especially white emitters.

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