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ORIGINAL ARTICLE

Synthesis, characterization and quantum yields of multichromic poly(azomethine)s containing carbazole unit

İsmet Kaya *, **Esra Kılavuz, Kevser Temizkan**

Polymer Synthesis and Analysis Laboratory, Department of Chemistry, Canakkale Onsekiz Mart University, Canakkale 17020, Turkey

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KEYWORDS

Poly(azomethine)s;
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Thermal degradation;
Fluorescence quantum yield

Abstract Poly(azomethine)s containing phosphor, silane and carbazole were synthesized with multiple stage and examined through different photophysical, electrochemical, and thermal behaviours. Following substances were synthesized as an initial step: N-hexyl-carbazole (CH) and N-hexyl-carbazolaldehyde (CHDA), via elimination reaction in argon media and N-hexyl-carbazole azomethine (CHA) via condensation reaction of CHDA with 4-aminophenol also poly(azomethine)s (P-Si-CHA, P-P-CHA) containing silane and phosphor via elimination reaction. The structures of synthesized compounds were confirmed by Fourier-transform infrared spectroscopy (FT-IR), Ultraviolet–visible spectroscopy (UV–Vis) and nuclear magnetic resonance spectroscopy (NMR). Electrochemical properties of compounds were examined with cyclic voltammetry (CV) technique. Fluorescence measurements were utilized to investigate the photochemical behaviors by photoluminescence (PL) analysis. CHA compound surprisingly presented multicolor emission behavior (when excited at 370, 420, 480, 540 and 580 nm, the solution emitted blue, green, yellow, orange and red lights, respectively) with relatively high quantum yield (19.9%) in DMF solvent. Additionally, thermal behaviors of all compounds were determined by TG and DSC techniques. Surface morphologies of polymers were imaged with scanning electron microscope (SEM).

1. Introduction

Carbazole is free of expensive chemical that can be enclosed with sort of conjugated units to produce new materials (Grazulevicius et al., 2003). Carbazole derivatives are convenient members of organic compounds having electronic structures for multipurpose applications such as organic light emitting diodes (OLEDs), (Jiang et al., 2012; Lee et al., 2013; Uoyama et al., 2012) electrochromic devices (Hsiao

* Corresponding author.
E-mail address: kayaismet@hotmail.com (İ. Kaya).
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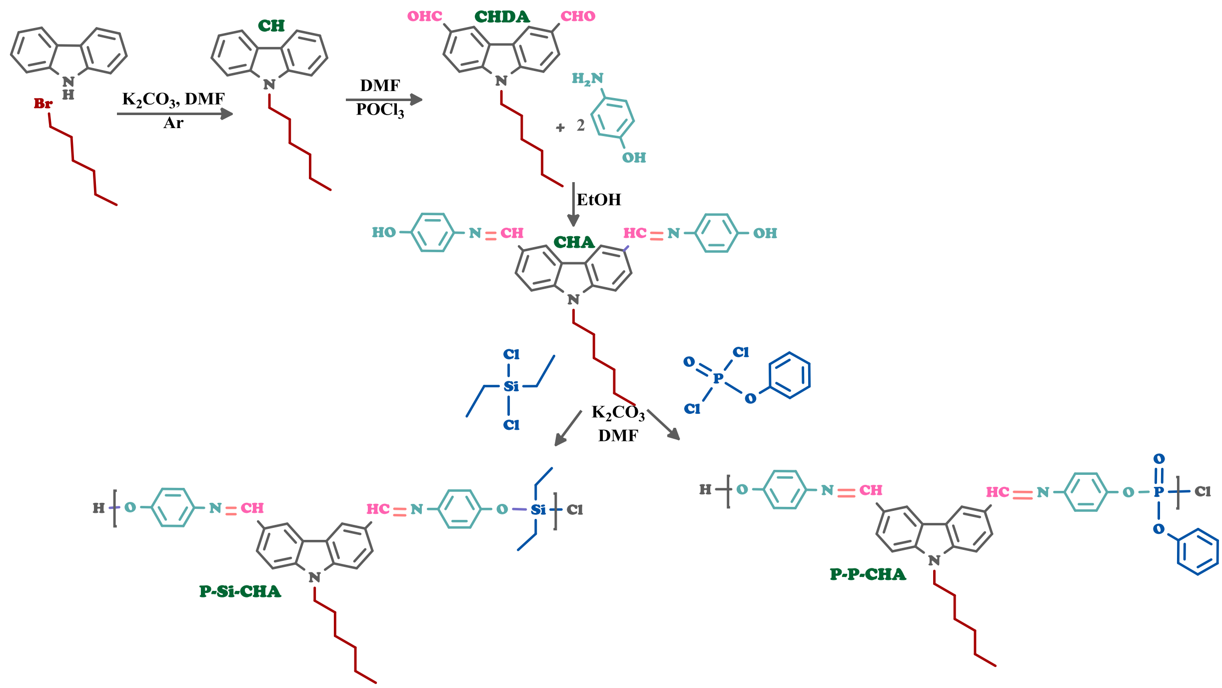


Scheme 1 Syntheses of carbazole derivative compounds and its polymers.

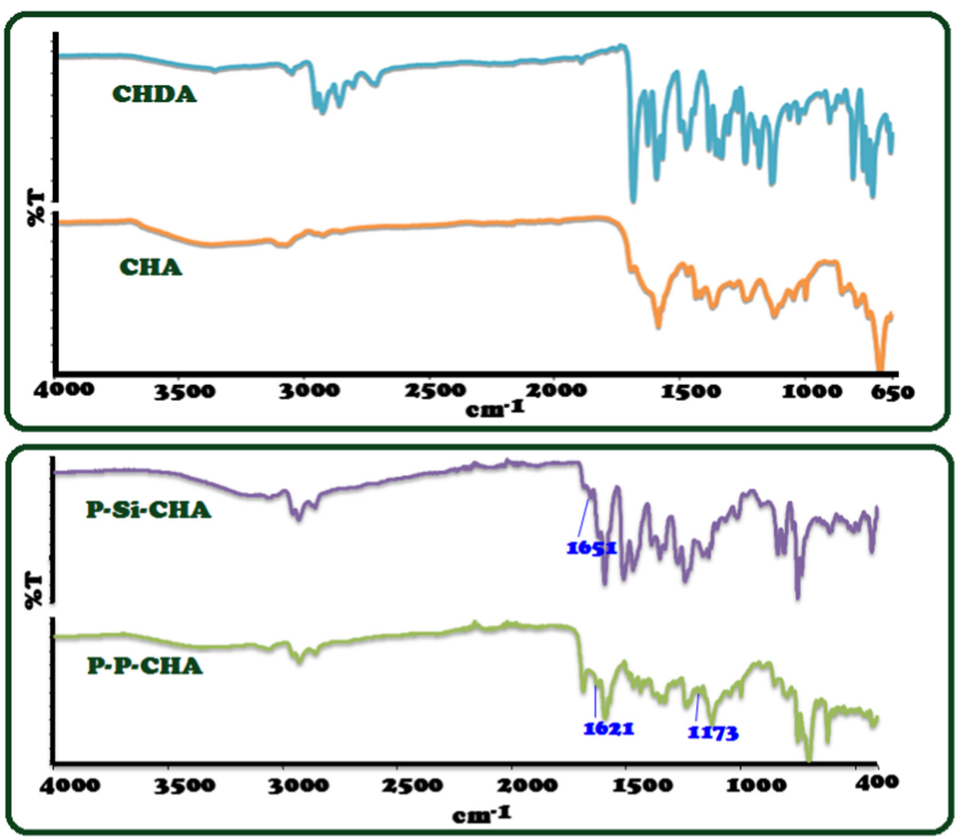


Fig. 1 FT-IR spectra of CHDA, CHA, P-Si-CHA and P-P-CHA.

and Lin, 2016), organic field effect transistors (OFETs) (Reghu et al., 2012; Chang et al., 2013), and solar cells (Tang et al., 2010; Li and Grimsdale, 2010; Lee et al., 2014). Most important properties of carbazole based compounds are high fluorescence quantum yields (Lai et al., 2011), high mobility (Reghu et al., 2012), high molar extinction coefficients (Cai

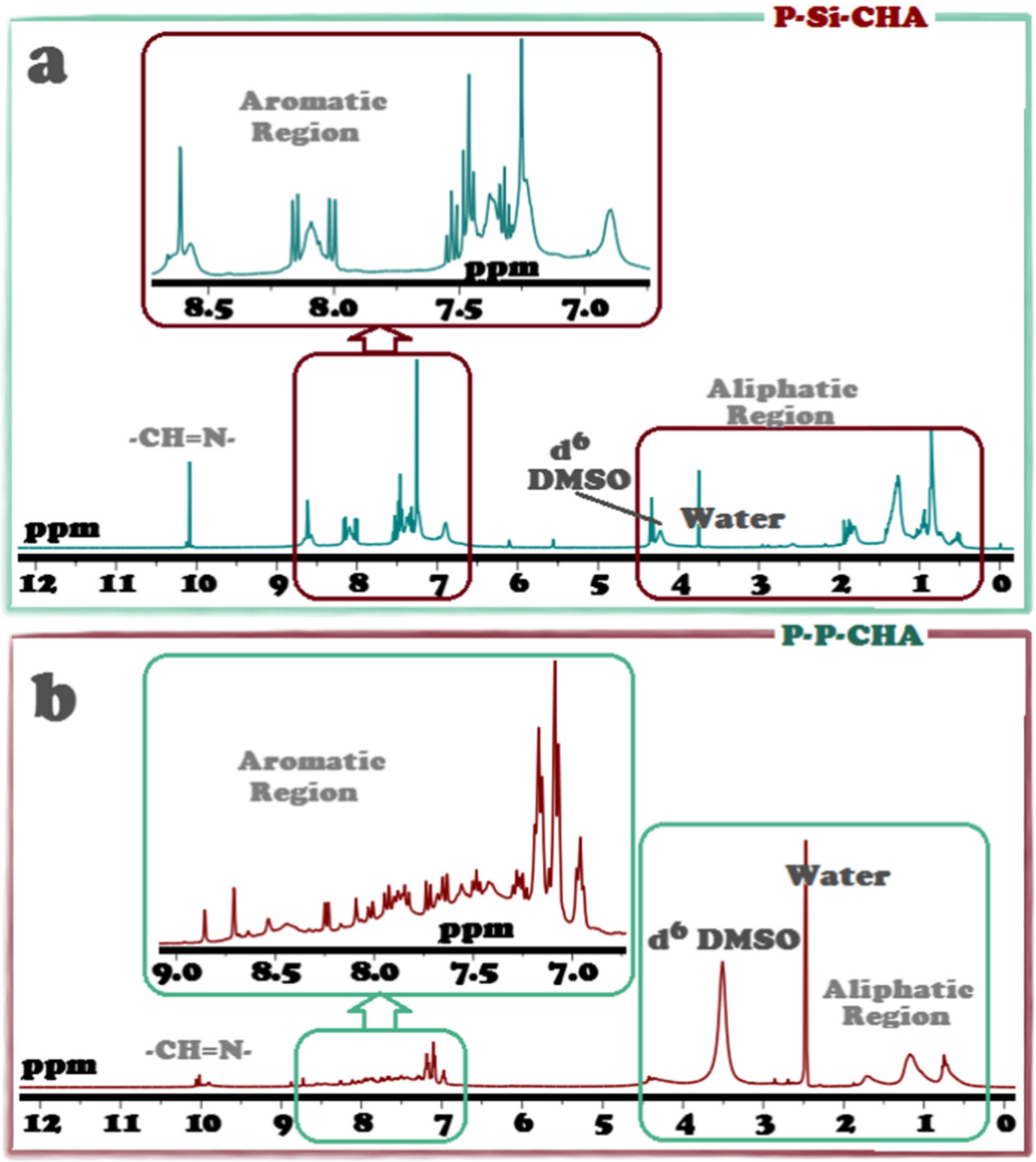


Fig. 2 ^{1}H NMR spectra of synthesized P-Si-CHA (a) and P-P-CHA (b).

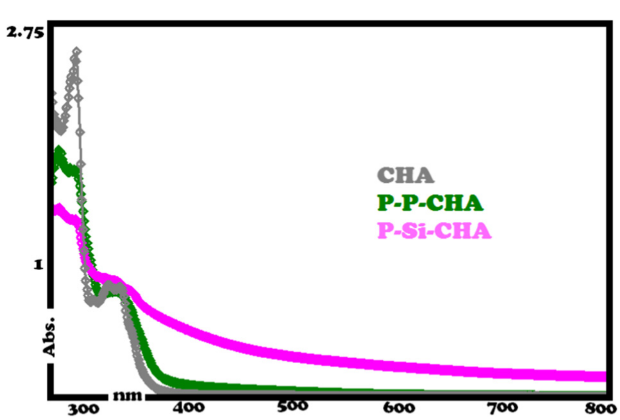


Fig. 3 UV–Vis spectra of CHA, P-Si-CHA and P-P-CHA.

Table 1 Optical band gap, λ_{max}, λ_{onset} values of the compounds.

Compounds	λ_{max} (nm)	λ_{onset} (nm)	E_g (eV)
CHA	290, 330	380	3.26
P-P-CHA	277, 333	510	2.43
P-Si-CHA	280, 337	650	1.90

et al., 2013), and ability for polymerization (Reghu et al., 2013), and other properties, such as self-assembly (Ding et al., 2013), aggregation for enhancement emission (Huang et al., 2014), and energy levels, modulated by changing the structure of molecule (Blouin et al., 2008).

Polymers containing carbazole moieties in the main chain or side chain have attracted much attention because of their unique property that allows multifarious electronic applica-

Table 2 Photoluminescence values of the CHA, P-P-CHA and P-Si-CHA.

Products	Concentration (mg/mL)	λ_{Ex} [a]	$\lambda_{max\ (Em)}$ [b]	I_{Em} [c]
CHA	0.025	370	494	654
P-P-CHA	0.025	320	362, 384	720
P-Si-CHA	0.025	320	355, 380	992

[a] Excitation wavelength for emission.
[b] Maximum emission wavelength.
[c] Maximum emission intensity.

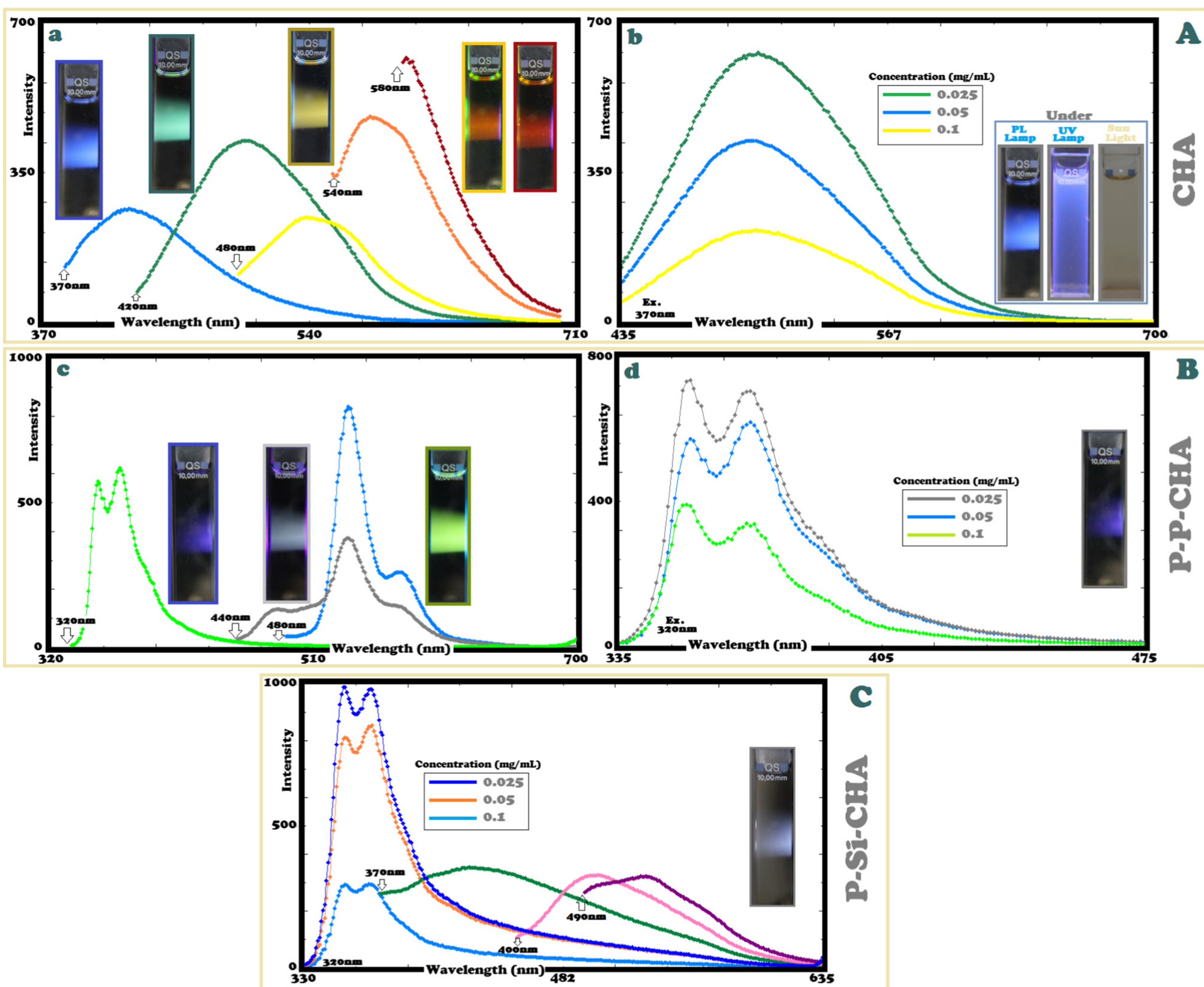


Fig. 4 Multichromic and fluorescence properties of synthesized compounds, A – PL spectra of CHA and their photographs, a – Different emission colors with different excitation wavelength (blue emission (370 nm/Slit 3), green emission (420 nm/Slit 3), yellow emission (480 nm/Slit 5), orange emission (540 nm/Slit 5) and red emission (580 nm/Slit 5)), b – Emission spectra of CHA with different concentrations in DMF solvent, B – PL spectra of P-P-CHA and their photographs, a – Different emission colors with different excitation wavelength (blue emission (320 nm/Slit 3), white emission (440 nm/Slit 3), green emission (480 nm/Slit 5), b – Emission spectra of P-P-CHA with different concentrations in DMF solvent C-PL spectra of P-Si-CHA and observed white emissions with different excitation wavelength (320, 370, 400 and 490 nm/Slit 3).

tions for example conductive, electroluminescent and electrochromic materials (Grazulevicius et al., 2003; Iraqi and Wataru, 2004a; Iraqi and Wataru, 2004b; Yi et al., 2007). At the same time, carbazole derivatives own electronic transportation features, adequately high triplet energy levels, and for these reason, oligo or poly(carbazole)s with 3(6),9-linkages are effective for phosphorescent materials (Ding et al., 2009; Lee et al., 2012; Xia et al., 2014; Wang et al., 2015; Dumur, 2015). With different synthetic techniques and substitution patterns, the physical and chemical features of poly-3,6-carbazoles/ poly-2,7-carbazoles can be fine for high performance materials and many electronic applications (Morin et al., 2005; Bloudin and Leclerc, 2008; Boudreault et al., 2010). As a class of conjugated polymers azomethine derivative polymers, which can be easily applied to various fields in material world, have high conductivity, phosphorescence, and photonic property in addition thermal stability (Morgan et al., 1987; Wojkowski, 1987; Yeakel et al., 1993; Yang and Jenekhe, 1995; Avcı et al., 2015).

Poly(silane)s containing Si backbone are use as active references for photo and electro luminescence materials (Ding et al., 2010; Okamoto et al., 2010; Singh and Katiyar, 2010; Ma et al., 2007; He et al., 2011). Moreover, poly(phosphazene)s containing phosphor atoms in polymer chain are especial form of hybrid inorganic and organic polymers with organic side groups. Poly(phosphazene)s with many useful properties have been introduced for multiple range of characteristics for tissue engineering practices. Poly(phosphazene)s are usually long molecular chained compounds. Besides, their physical and chemical properties are considerably allied with the nature and composition of the substituted groups (Jiang et al., 2006; Allcock, 1972).

In this study, we synthesized derivatives of conjugated poly (azomethine)s containing phosphor, silane and carbazole. The structures and characterization of compounds were investigated by using FT-IR, UV–Vis, NMR, TG/DTG, DSC, CV, photoluminescence/fluorescence/multichromic (PL) and surface (SEM) measurements.

2. Experimental

2.1. Chemicals

Carbazole, 1-bromohexane,phosphoryl chloride, 4-amino phenol, diphenyl phosphoryl chloride, dichlorodimethylsilane were supplied by Sigma Aldrich and 3,5-bis(bromomethyl) toluene were supplied by Alfa Aesar. Potassium carbonate (K_2CO_3), dimethylformamide (DMF), methanol (MeOH), ethanol (EtOH), chloroform, ethyl acetate, hexane, dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), acetonitrile were purchased from Merck Chem. Co. (Germany).

2.2. Synthesis of poly(azomethine)s containing silane, phosphor and azomethine groups

Synthesis process of this study is shown in Scheme 1 at the fourth step. Synthesis of the materials through using elimination and condensation reactions shown respectively in the Scheme 1as follows: CH in the first step, CHDA in the second step, CHA in the third step, and poly(azomethine)s containing silane, phosphor and carbazole (P-Si-CHA, P-P-CHA) in the last steps.

CH and CHDA were synthesized with using information of previous studies (Allcock, 2003; Ostrauskaite et al., 2002). CHDA (0.02 mol) which has already been obtained in the previous step was dissolved in EtOH (15 mL) in the third step.

An amount of 0.04 mol of 4-amino phenol, dissolved in EtOH (5 mL), was added to this mixture and heated (70 °C, 6 h), slowly. This mixture was filtered, recrystallized from acetonitrile and dried in vacuum oven at 50 °C for 24 h (Goswami et al., 2013). For the fourth step, CHA (0.2 mol) which had obtained in previous step, was dissolved in DMF (20 mL). Then transferred into two different 250 mL-flask.0.02 mol of K_2CO_3 was dissolved in DMF (5 mL) and added to the prepared mixture. Given mixture was heated to 60 °C for 1 h under argon gas. Amount of 0.02 mol of diphenyl phosphoryl chloride and dichloro-dimethyl silane was dissolved in DMF (30 mL) and they were added to signified mixtures, respectively. The temperature improved at 150 °C and the reaction mixture was stirred into argon gas for 16 h and left one-night for the consummation of the reaction. These reaction solutions were decanted into 200 ml of iced water and the settled pure products were accumulated (Kaya and Kılavuz, 2015). The obtained polymers were washed with methanol (25 mL). These products were dried in vacuum oven at 50 °C for 24 h.

2.3. Characterization techniques

Ultraviolet–Visible absorption spectra of the compounds were analyzed by AnalytikJena Specord 210 Plus double beam spectrophotometer between 240 and 800 nm. Solutions of synthesized compounds were prepared by dissolving 2–3 mg in 10 mL DMF at 25 °C for UV–Vis Spectroscopy analysis. Fourier transform infrared (FT-IR) spectra of obtained compounds were analyzed by PerkinElmer FT-IR-FIR Spectrum between 400 and 4000 cm^{-1}. ^{1}H-NMR spectra (Agilent 600 MHz Premium COMPACT NMR Magnet) were obtained by DMSO-$_{d6}$ as a solvent at room temperature. Fluorescence spectra were obtained with Shimadzu RF-5301PC spectrofluorofotometer. TG-DTA measurements were taken in PerkinElmer Diamond Thermal Analysis system between 20 and 1000 °C (heating rate 10 °C min^{-1}) under nitrogen. DSC analysis were realized from heating the polymer under N_2 purging with PerkinElmer Pyris Sapphire. For the tests of TGA and DSC, ceramic capsule and aluminum pan were used, respectively, and the amount of each sample was selected as 10 mg. Number of average molecular weight (M_n), weight average molecular weight (M_w) and polydispersity index (PDI) were obtained and calculated with Gel Permeation Chromatography-Light Scattering (GPC-LS) instrument of Malvern Viscotek GPC Dual 270 max. SEM photographs/images of synthesized polymers were obtained as using Jeol JSM-7100F Scotty instrument.

3. Results and discussion

3.1. Spectral analysis of compounds

FT-IR spectra of synthesized compounds which were CHDA, CHA, P-P-CHA and P-Si-CHA are given in Fig. 1. FT-IR spectrum of CHDA has a specific peak at 1681 cm^{-1} related to aldehyde vibrations. FT-IR spectrum of CHA has two specific peak at 1628 and 3295 cm^{-1} related to imine (—CH=N—) and —OH vibrations, respectively. According to FT-IR spectra of P-P-CHA and P-Si-CHA polymers have imine peak at 1621 and 1651 cm^{-1} and also P-P-CHA has a

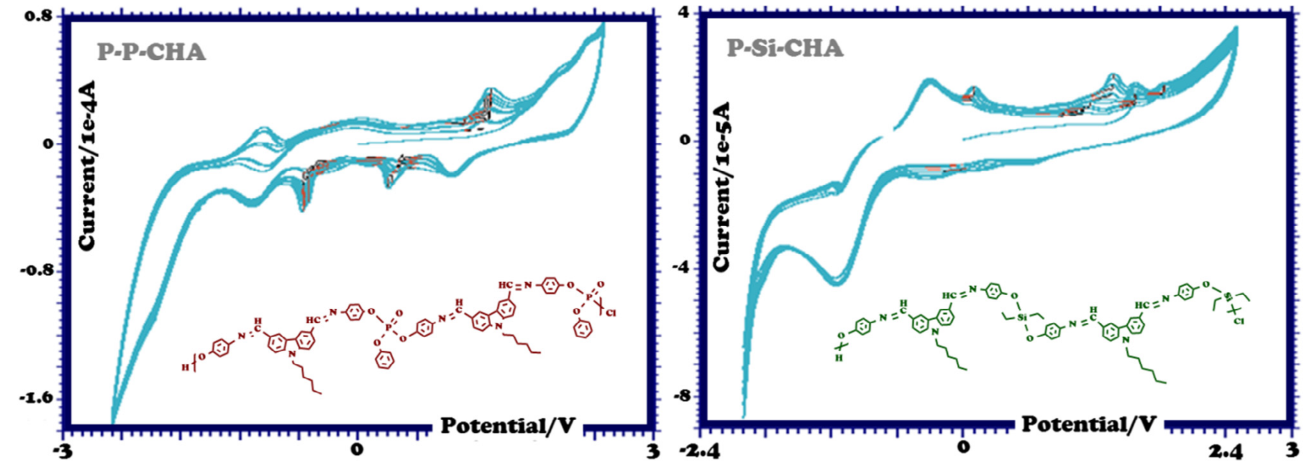


Fig. 5 Cyclic voltammograms of P-P-CHA and P-Si-CHA (scan rate: −3 to 3 V/s).

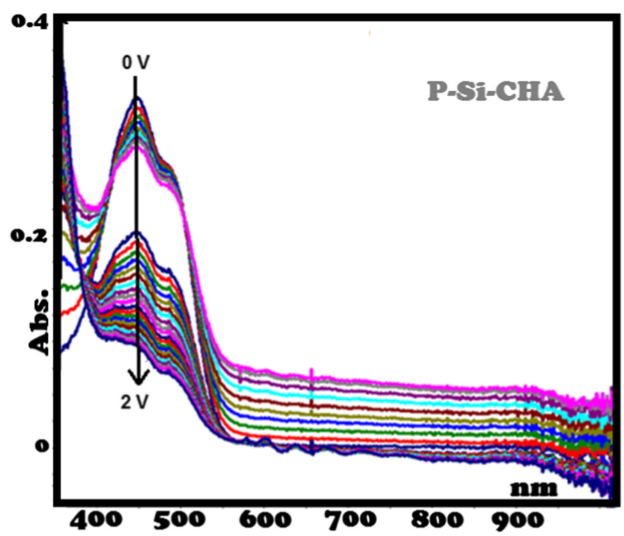


Fig. 6 UV–Vis spectra of P-Si-CHA (decreasing form between 0 V and 2 V).

peak at 1173 cm^{-1} related to —P=O vibrations. All specific peaks of compounds are verified structures in Fig. 1.

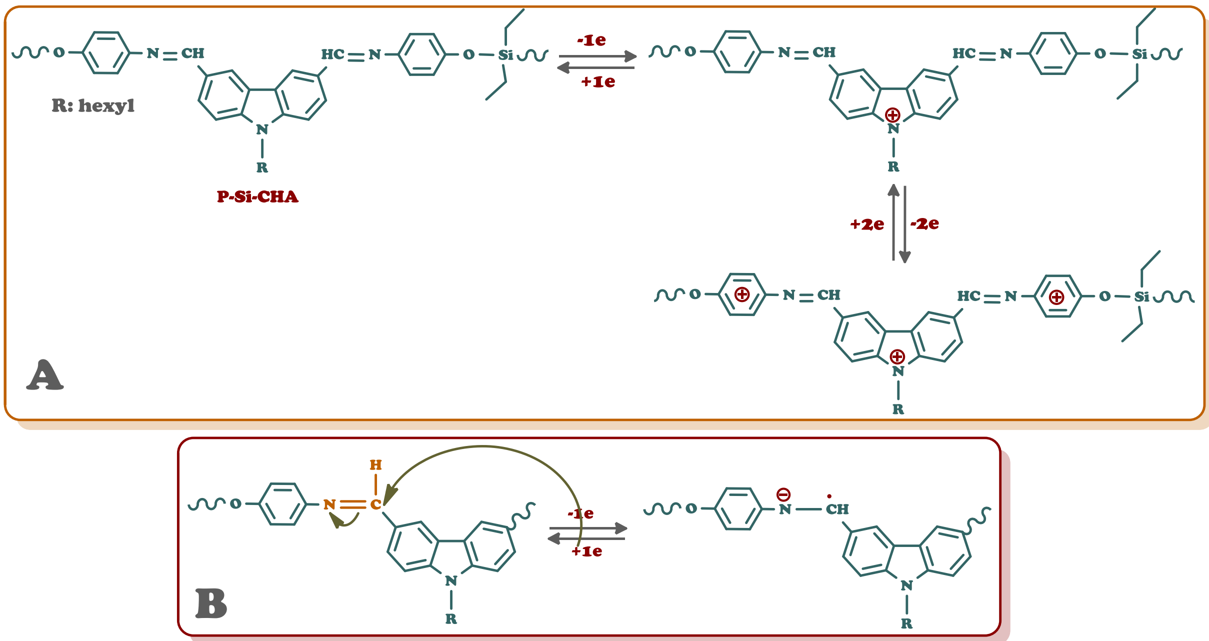
^{1}H NMR results of synthesized carbazole polymers are demonstrated and summarized in Fig. 2. Imine peaks are seen at 10.00 and 10.08 ppm for P-P-CHA and P-Si-CHA, respectively. In addition, aromatic and aliphatic protons of P-P-CHA and P-Si-CHA are seen between 8.86–6.96 ppm, 4.40–0.73 ppm and 8.63–6.89 ppm, 4.32–0.85 ppm, respectively. According to these ^{1}H NMR results, the structures of polymers are verified.

UV–Vis spectra and obtained data from these spectra of the synthesized compounds CHA, P-P-CHA and P-Si-CHA are given in Fig. 3 and Table 1, respectively. According to CHA spectra, CHA has two absorption band. First absorption band is between 280 and 320 nm due to $\pi \rightarrow \pi^{*}$ transition from benzene conjugation and second absorption band is between 320 and 380 nm $\pi \rightarrow \pi^{*}$ transition from azomethine conjugations (Kaya et al., 2013). According to P-P-CHA spectra containing P atoms, P-P-CHA has two absorption bands. First absorption band is between 270 and 315 nm due to $\pi \rightarrow \pi^{*}$ transition from benzene conjugation and second absorption band is between 315 and 515 nm $\pi \rightarrow \pi^{*}$ transition from azomethine conjugations. According to containing Si atoms, P-Si-CHA spectra, P-Si-CHA has two absorption bands. First absorption band is between 270 and 320 nm due to $\pi \rightarrow \pi^{*}$ transition from benzene conjugation and second absorption band is between 320 and 650 nm $\pi \rightarrow \pi^{*}$ transition from azomethine conjugations (Aldea et al., 2016).

According to gel permeation chromatographic (GPC) analysis, M_w and PDI values of P-P-CHA and P-Si-CHA were calculated as 21,900 Da, 18,400 Da and 1.38 and 1.23, respectively. The repeated unit numbers of P-P-CHA and P-Si-CHA were found as 35 and 32, respectively. According to these results, polymerization of compound containing aliphatic silane group was less than phosphazene unit.

3.2. Fluorescence properties

PL spectra of synthesized compounds were recorded by Shimadzu RF-5301PC spectrophotometer. Photoluminescence values of the CHA, P-P-CHA and P-Si-CHA are given in Table 2. Standard solution sample for all analysis was prepared by using fluorescein in 0.1 M NaOH solution Fig. 4 demonstrates that PL spectra and photographs of synthesizes compounds in selected solvent DMF. It is seen that Fig. 4A-a, CHA has five different emission color at different excitation wavelengths. That means CHA was shown multicolor emission properties in DMF. CHA exhibited five strong emission peaks with different colors and quantum yields that blue/9.8%, green/19.9%, yellow/2.1%, orange/2.8% and red/1.7%, when excited at 370 (slit 3), 420 (slit 3), 480 (slit 5), 540 (slit 5) and 580 (slit 5) nm respectively (Kaya et al., 2013). In Fig. 4A-b, it' seen that PL spectra of CHA with different concentration when excited at 370 nm and solution images under PL, UV and sunlight. It is seen that Fig. 4B-c, P-P-CHA compound has three different emission color at different excitation wavelengths. That means P-P-CHA shows multicolor emission properties in DMF. P-P-CHA exhibited three strong emission peaks with different colors and quantum yields that blue/18%, white/6.3%, green/4.4%, when excited at 320 (slit 3), 440 (slit 3), 480 (slit 5), nm respectively (Doğan et al., 2016). In Fig. 4B-d, it is seen that PL spectra of P-P-CHA with different concen-



Scheme 2 The oxidation reaction mechanisms of the P-Si-CHA polymer (A) and the electrochemical reaction mechanisms of the polymers (B).

tration when excited at 320 nm and solution image under PL light. It is seen that Fig. 4C, P-Si-CHA compound is white emission polymer. When it was excited at 320, 370, 400 and 490 nm, solution depicted white emission. Quantum yields of P-Si-CHA were calculated as 3.2%, 4.6%, 2.8% and 2.2% (Maity et al., 2014; Xu et al., 2016) when it was excited at 320, 370, 400 and 490 nm, respectively. In the same time, Fig. 4C demonstrates that PL intensity of compound with different concentration. CHA and P-P-CHA are good blue and green light emitters with 19.9% and 18% quantum yields (Miwa et al., 2017). They can be used in blue or green organic light emitting diodes because of their high quantum yields. They can be used in blue or green organic light emitting diodes because of their high quantum yields. These materials that have high quantum yields are useful for bioimaging technologies, biosensors, drug delivery systems/biomedicine, anticancer drugs, and biomimetic membrane materials in aqua solutions (Zhu et al., 2016; Tang et al., 2010; Muhammad et al., 2011).

3.3. Electrochemical properties

Specific cyclic voltammograms (CV) of synthesized carbazole polymers are seen in Fig. 5, during process of electrochemical polymerization. According to these voltammograms, it is seen that reversible oxidation and reduction potentials for P-P-CHA at nearly 0.97, 1.56, 2.31 V and −1.95, −1.05, −0.59 V and for P-Si-CHA at nearly 1.57, 1.70, 2.00 V and −1.72, −1.10 V, respectively. Increase of these all oxidation and reduction peaks were verified that polymers were accumulated on electrode surface and electro polymerization was occurred (Yıldırım et al., 2016).

In order to reveal optoelectrochemical properties of the P-P-CHA and P-Si-CHA polymers, their spectral behaviors and characteristics should be evidenced by their electronic absorption spectra underperformed potentials. In addition, spectroelectrochemical analyses were occurred with different potentials in 0.1 M AN/$LiClO_4$ solution. Fig. 6 shows the changes in the absorption spectra of P-Si-CHA at various performed potentials. Intensities of polymers π-π transitions attenuated and reduced shape of P-Si-CHA owns an absorption maximum at 465 nm, which subtends to diminishing from 0.0 V to 2.0 V. The oxidation and electrochemical reaction mechanisms of P-Si-CHA polymer are given in the Scheme 2A and B, respectively. There is similar the oxidation and electrochemical reaction mechanisms in for P-P-CHA polymer.

3.4. Thermal properties

Thermal (TG/DTG-DSC) curves and thermal degradation results of synthesized P-P-CHA and P-Si-CHA compounds

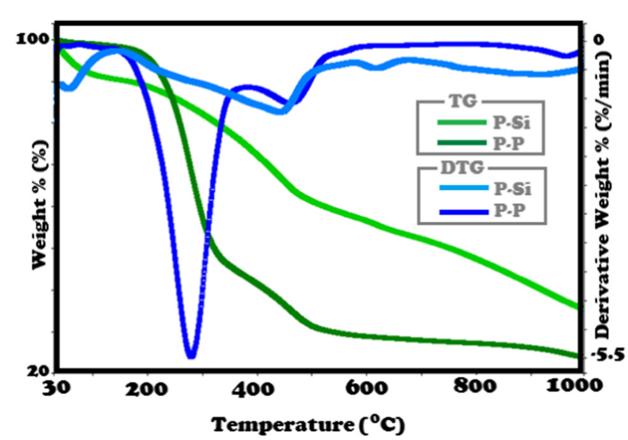


Fig. 7 TG and DTG curves of the synthesized P-P-CHA and P-Si-CHA.

Table 3 Thermal data of synthesized polymers.

Compounds	T_{on}[a]	$W_{max}T$[b]	T_{20}[c]	T_{50}[d]	% Char at 1000 °C	Loses of Solvent (%)	DSC T_g (°C)[e]
P-P-CHA	227	280,461	262	330	24.41	1.50	177
P-Si-CHA	280	444,620	324	750	41.54	8.81	193

[a] The onset temperature.
[b] Maximum weight temperature.
[c] 20% weight loss.
[d] 50% weight loss.
[e] Glass Transition Temperature.

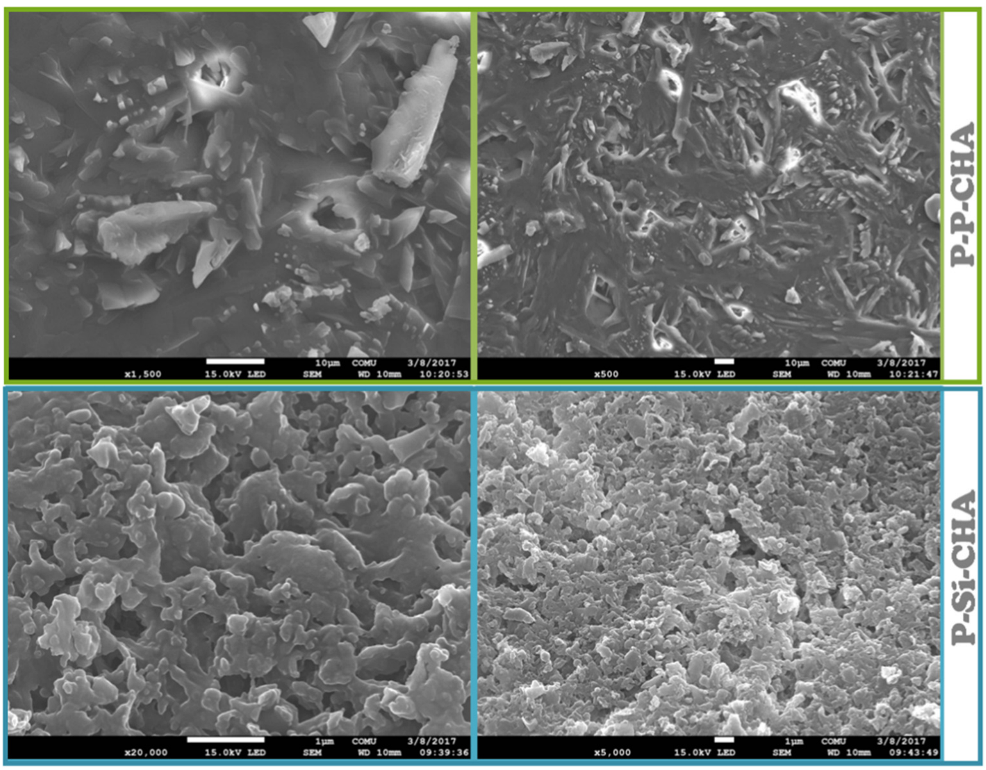


Fig. 8 FE-SEM images of polymers (P-P-CHA/green framed and P-Si-CHA/blue framed).

are given in Fig. 7and Table 3, respectively. According to calculated results from these curves, P-P-CHA and P-Si-CHA polymers were demonstrated two decomposition steps. According to TG curves of P-Si-CHA and P-P-CHA, onset temperatures were found as 280 and 227 °C, respectively. The first and the second decomposition steps of P-P-CHA were observed between 130–382 °C and 382–1000 °C and weight losses of their existing amount were 56.51% and 19.08%, respectively. The first and the second decomposition steps of P-Si-CHA were observed between 150–575 °C and 575–1000 °C and weight losses of their existing amount were 34.15% and 24.31%, respectively. The mass losses of P-P-CHA and P-Si-CHA were found as 1.50% and 8.80% at lower temperature between 30–130 °C and 30–150 °C, respectively. These losses may be due to the volatilization of water or organic solvent. Both onset temperature and char% of P-Si-CHA was higher than P-P-CHA compound. Thermal properties of imine polymers containing silane group had examined in the literature (Kaya et al., 2017). The glass transition temperature (T_g) and ΔCp values of P-Si-CHA and P-P-CHA were found an as 190 °C and 0.032 J/g °C; 177 °C and 0.028 J/g °C, respectively, from DSC measurements.

3.5. Surface properties

FE-SEM images of polymers belonging to P-P-CHA/green framed and P-Si-CHA/blue framed are shown in Fig. 8. According to SEM photographs of these polymers, P atoms containing in main chain/ P-P-CHA has inhomogeneous and ragged surface also Si atoms containing in main chain/P-Si-CHA has homogeneous dispersion, elliptical particles and overall image like spongiform. The coatings can be used for biomaterials thanks to be containing biocompatible Si and P atoms with certain modification as in previous as in previous studies (Zhou et al., 2016).

4. Conclusion

In conclusion, we synthesized phosphor, silane and azomethine derivative conjugated poly(azomethine)s with multiple stage synthesis. In the first step N-hexyl-carbazole (CH), in the second step N-hexyl-carbazole aldehyde (CHDA), in the third step N-hexyl-carbazole azomethine (CHA) and in the last steps poly(azomethine)s (P-Si-CHA, P-P-CHA) containing silane and phosphor were synthesized by elimination and condensation reactions. The obtained compounds were structurally verified through using FT-IR and UV–Vis techniques. In addition, electropolymerization performed with obtained two poly(azomethine)s by CV and UV–Vis analysis. According to result of thermal analysis, P-P-CHA and P-Si-CHA polymers have high thermal character; onset temperature and % char (at 1000 °C) of synthesized compounds, like 237 and 284 °C; 24% and 36%, respectively. In addition, these compounds especially CHA have ultra-high fluorescence properties, multichromic character with different color emissions and novel quantum yields. P-Si-CHA which contains Si atoms emits only white light when excited at 320, 370, 400, 490 nm and has maximum 4.6% quantum yield (excited 370 nm). P-P-CHA demonstrates three color emission; blue, white and green when excited 320, 440, 480 nm, respectively. In addition, P-P-CHA has maximum quantum yield (with blue light) that is so high; 18% when excited at 320 nm. CHA demonstrates five color emission; blue, green, yellow, orange and red when excited 370, 420, 480, 540 and 580 nm, respectively. In addition, CHA has maximum quantum yields (with blue and green lights) which are so high; 9.8% and 19.9% when excited at 370 and 420 nm, respectively. Synthesized three compounds can be useful and good candidate for electrochromic device and organic light emitting diodes (OLEDs).

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