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ORIGINAL ARTICLE

Optical Properties of Azo-Benzothiazole Side Chain Liquid Crystalline Polymers: Effect of Solvents, Substituents and Temperatures

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Abstract

The optical properties of a series of side chain liquid crystalline polymers (P1–P3) containing azo-benzothiazole mesogen with different terminal substituents (–H, $–CH_3$ and $–OCH_2CH_3$) in four organic solvents of varying polarity have been investigated by absorption and fluorescence spectral analysis. Solvatochromic studies of P1–P3 did not show any regular variation on the absorption and emission intensities with changing the polarity of solvent. Theoretical studies were performed based on different solvent correlation methods such as Dimroth-Reichardt and Kamlet-Taft methods to investigate the solute-solvent interactions. Both absorption and emission maxima of investigated polymers were bathochromically shifted with the replacement of sixth position hydrogen atom by electron donating groups in benzothiazole moiety. The emission intensities of the studied polymers showed decreasing trend with increasing temperature.

Keywords Azo-benzothiazole · Side chain liquid crystalline polymer · Solvatochromism · Thermochromism

Introduction

Organic fluorescent materials that exhibit a fluorescence response to external stimuli such as pressure and heat have received tremendous attention because of their potential practical applications as sensors [1], in optical recording [2] photonics [3] and optoelectronic devices [4, 5] In recent years, these stimuli-responsive photoluminescent materials, including those that are thermochromic, photochromic, mechanochromic, solvatochromic and electrochromic, have been developed widely as they have been extremely "smart" materials, since the external force can be easily controlled or modulated [6].

Solvatochromic molecules are a class of compounds which show spectral shift (absorption and/or emission maxima) with changing polarity of its surrounding environment. Both of intra- and intermolecular interactions between the solute and solvent exhibit a profound effect on the geometric, electronic and vibrational properties of aromatic molecules in their excited states [7]. These interactions can be classified into: (i) non-specific solute-solvent interaction caused by polarity-polarizability effects and (ii) specific solute-solvent interaction such as hydrogen bonding or electron donor-acceptor interaction [8].

Several theoretical models such as Lippert-Mataga, Weller's, Rettig's, McRay's plots, Kamlet-Taft [9], and Dimroth-Reichardt's methods [10] have been proposed to describe the preferential solvation phenomenon. Jiang et al. [11] reported that the variation of Stock shifts with solvent polarity on naphthalimide-carbazole dyes using Lippert-Mataga plot was mainly due to the large change in dipole moment upon excitation. In another work, Alphonse and co-workers [12] described that both absorption and emission spectra of their investigated compounds were red shifted by controlling hydrogen bond donor strength (α) of solvent and the theoretical calculations were made by using Kamlet-Taft method.

Besides solvatochromism, a study of thermochromic effect on the optical and electrical properties would also result to an intriguing and interesting phenomenon that could make the materials useful for electronic and optoelectronic devices [13]. The underlying theory to the spectral shifts involves

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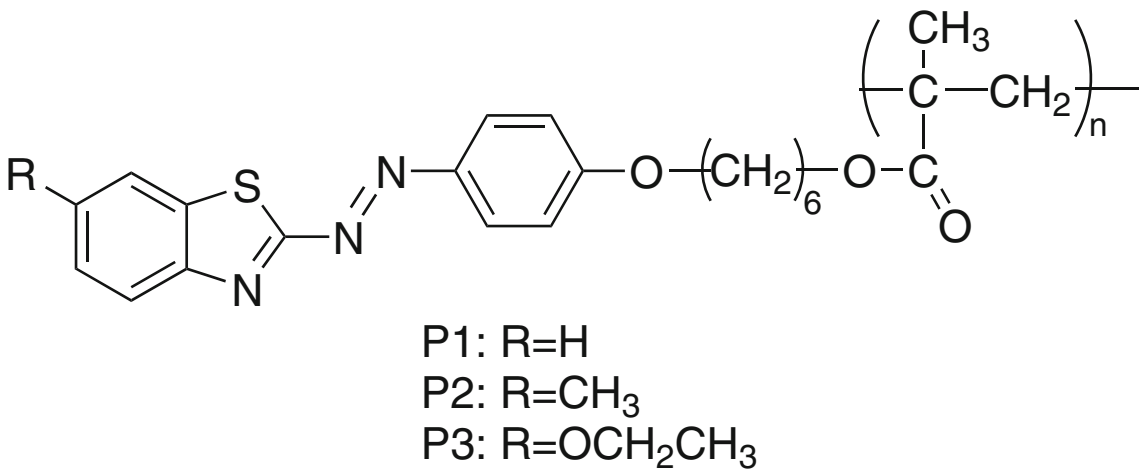

Fig. 1 Structural formulae of polymers P1–P3

various factors including the mechanism of temperature effects on the energy of electronic states, dipole moments of excited molecules in solvents [14], changes in refractive index of the solution [15], polarizability of medium [16] and solvents' dielectric constant [17].

In the last few decades, liquid crystalline polymers containing azo-chromophore have been extensively studied due to their unique photochromic nature of azo group. These unique features can be exploited for various technological applications including optical data storage, nonlinear optical (NLO) materials, photo-switching devices, organic light-emitting devices (OLEDs) [18], liquid crystal displays [19], semiconductor materials [20] and so on. Although the effect of solvent [21–24] and temperature [25, 26] on photophysical studies of organic compounds have extensively been investigated, but to the best of our knowledge, research on the solvatochromic and thermochromic behavior of side chain liquid crystalline polymers (SCLCPs) have rarely been reported.

In our previous work [27, 28], we described the synthesis, spectroscopic characterization, and various physical properties of a series of SCLCPs having azo-benzothiazole moiety. Herein, we report the effect of solvent and temperature on the electronic absorption and fluorescence spectra of the polymers. The solvent effects were then analyzed using Dimroth-Reichardt and Kamlet-Taft solvent scales. In addition, the substituent and temperature effects on both absorption and emission spectra of P1–P3 were also evaluated by UV-vis and fluorescence spectroscopies.

Experimental Section

Materials

Tetrahydrofuran (THF), chloroform ($CHCl_3$), chlorobenzene and dichloromethane (DCM) were purchased from Merck and were used without further purification. The synthesis and characterization of the studied polymers, P1–P3 were described in our previous report [29]. The purity of P1–P3 was estimated to be ≥98% according to ^{1}H NMR, ^{13}C NMR, and thin layer chromatography. The structural formulae of the investigated polymers, P1–P3 are illustrated in Fig. 1.

Instrumentations

Absorption and emission spectra were recorded on Cary 60 UV-vis and Shimadzu RF-5301 PC spectrophotometer, respectively. Polymers, P1–P3 were dissolved with a concentration of 1×10^{-6} M in four organic solvents having different polarity parameters. The effect of temperature on emission spectra of polymers was studied from 20 °C to 55 °C using Photon Technology International EL-1000 spectrofluorometer equipped with temperature controller. In all the experiments, samples were measured in a 1 cm path-length quartz cell.

Table 1 Absorption and emission maxima of P1–P3 in selected solvents

Solvents		$CHCl_3$	THF	Chlorobenzene	DCM
π^*[a]		0.58	0.58	0.71	0.82
α [a]		0.44	0.00	0.00	0.13
β [a]		0.00	0.55	0.07	0.10
$E_T(30)$ [a]		39.10	37.40	36.80	40.70
ε [a]		4.81	7.52	5.62	8.93
P1	λ_{abs} (nm)	398	397	401	395
	λ_{em} (nm)	452, 481	461	452, 504	447
P2	λ_{abs} (nm)	403	401	409	396
	λ_{em} (nm)	491	474	454, 505	454
P3	λ_{abs} (nm)	424	423	428	423
	λ_{em} (nm)	486, 513	482	511	483, 525

[a] was taken from [9, 10, 30]

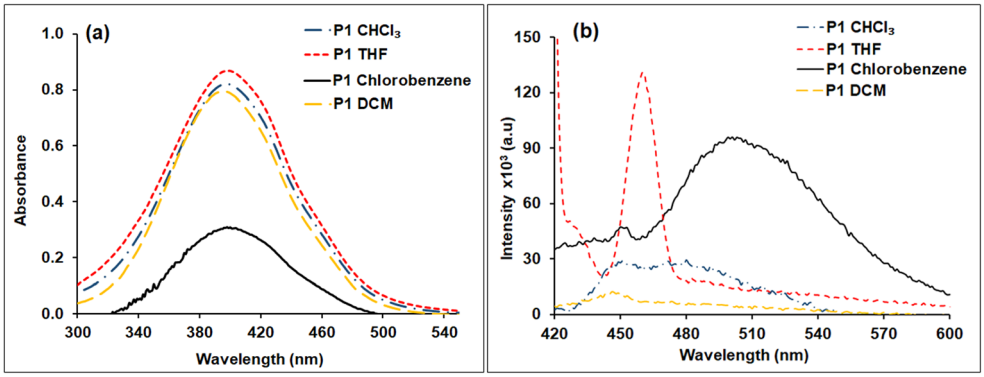

Fig. 2 (**a**) Absorption and (**b**) emission spectra of P1 in four selected solvents

Results and Discussion

Solvent Effect

The absorption and emission spectra of P1–P3 were recorded in selected organic solvents with a concentration of 1×10^{-6} M at room temperature and the results are shown in Table 1. The solvents are arranged in an ascending solvent polarizability: $CHCl_3$, THF, chlorobenzene, and DCM [9, 10, 30].

Figure 2 (a) shows UV-vis absorption spectra of P1 in different solvents. According to Fig. 2 (a), absorption bands as well as absorption maxima of P1 change in an irregular fashion with varying solvent polarity. This inconsistency of plot indicates that polymer P1 displays a polytonic character in all the employed solvents. The polytonic behavior of the solute-solvent interaction cannot be explained based on reciprocal polarization effects because wavelength does not vary proportionally change by the change of media dielectric constant as observed in benzoic acid liquid crystal [31].

Bozic et al. [32] reported a similar behavior of azo pyridone dyes in which the absorption spectra did not change significantly in the studied solvents and they could not correlate with the polarity of solvents. Alizadeh et al. [33] considered this phenomenon is due to combination of several solvent characteristics such as polarity, basicity, and H-bond-accepting ability. Likewise, P2 and P3, show similar behavior as P1 and their absorption spectra can be found in Supporting Information, Section A.

Table 2 Data of linear correlations between the absorption wavenumbers v_a (cm^{-1}) of P1–P3 and $E_T(30)$ values (cm^{-1}) for n different solvents, according to $v_a = v_0 + S.\ E_T(30)$,with r as correlation coefficient

Polymers	v_o(×10³ cm^{-1})	S(cm^{-1})	r	n
P1	22.370	0.072	0.804	4
P2	18.870	0.156	0.826	4
P3	21.753	0.047	0.632	4

To explain the effect of solvent polarity on the emission spectra of P1–P3, the emission maxima of the longer wavelength of the polymers in different solvents were taken as reference. As depicted in Fig. 2 (b) and Supporting Information, Section B, the emission maxima of P1–P3 displays a polytonic character ranging from 447 to 525 nm, attributing on the change of solvent polarity independently. Similar observation has been observed in some azo dyes [33]. Polymers, P1–P3 exhibit two emission bands in certain chlorinated solvents: (i) shorter wavelength emission bands around 452–486 nm and (ii) longer wavelength emission maxima about 481–525 nm. The first emission peak corresponds to the π-π* transition of the benzenoid rings while the latter peak may attribute to the benzothiazole moiety. The poor fluorescence intensities of P1–P3 in DCM and $CHCl_3$ can be linked to small concentration of isomers that inhibit proper interaction of the solute and solvent due to large intermolecular distances [34].

Correlation with Solvent Polarity Scales

(i) Dimroth-Reichardt- solvent scales, $E_T(30)$

In order to investigate the solute-solvent interactions, an empirical solvent polarity parameter, $E_T(30)$, has been described in this section [35]. The non-linear correlation of maximum absorption energy with Dimroth-Reichardt-solvent $E_T(30)$ parameter are tabulated in Table 2 and displayed in Fig. 3. The application of $E_T(30)$ values shows non-linear relations indicating that the change in the position of the charge transfer (CT) band is not due to dielectric properties only but can be considered due to the different factors governed by the various solvent parameters. These factors may be additive, counter-acting or may even cancel each other out [36]. Additionally, the results of correlation between v_{max} and $E_T(30)$ values can be explained by considering the fact that the $E_T(30)$ parameter represents the contribution of dipolarity/polarizability and acidity, but not basicity [37]. Thus, the plot elucidates that the specific solute-solvent interaction plays an important role in determining the spectral

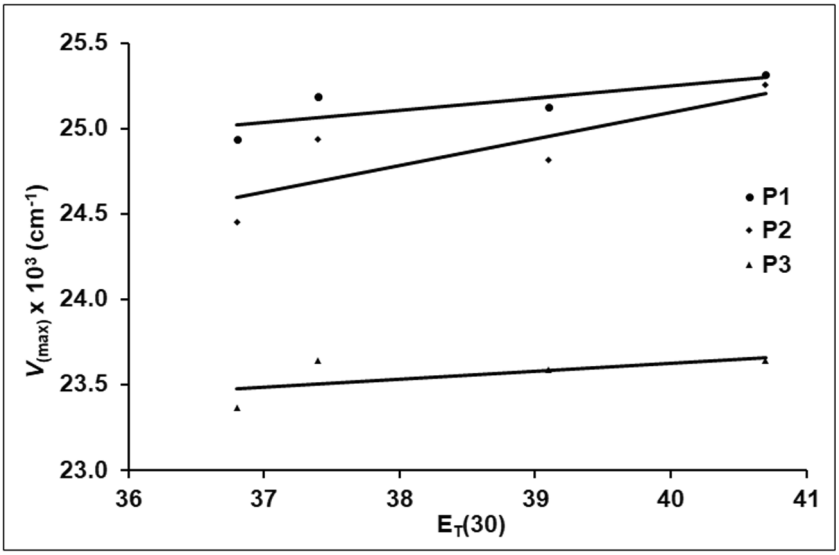

Fig. 3 Plot of absorption maxima (ν_{max} in 10^3 cm^{-1}) versus $E_T(30)$ for P1–P3 in Chlorobenzene (36.80), THF (37.40), Chloroform (39.10) and DCM (40.70)

position. This involves changes in the solution energies of the ground and excited states as well as solute-solvent hydrogen bonding [38].

(ii) Kamlet-Taft solvent scales

Kamlet-Taft's solvatochromic comparison method was used to analyze the solvatochromic behavior of P1–P3 at their maximum absorption band. This method rationalized solvent effects in terms of a linear combination, measuring specific interaction that is local to the solvation shell in the dissolved solvatochromic solute by quantifying the solvent's hydrogen bond donor ability α, hydrogen bond acceptor ability β, and the dipolarity/polarizability π* [39]. These solvatochromic parameters are used in linear solvation energy relationship of the general form in Eq. 1.

$$\nu_{\max} = \nu_o + s\pi^* + a\alpha + b\beta \quad (1)$$

where ν_o is the correlated wavenumber; s, a and b coefficient in Eq. 1 measure the relative susceptibilities of the absorption frequencies to the indicated solvent parameters [40]. The correlations of the absorption frequencies ν_{max} for the polymers have been realized by means of multiple linear regression analysis and the results are presented in Table 3.

From the analysis of the absorption frequencies according to Kamlet-Taft (Eq. 1), it was found that the positive sign of s coefficient for P1–P3 indicates a hypsochromic shift (blue shift) with increasing solvent polarity [41]. The percentage contribution of solvatochromic parameters for all polymers show that the negative solvatochromism were determined mainly by dipolarity/polarizability due to the greater coefficient of s term. The correlation also revealed that the solvent hydrogen bond donor capacity (HBD) is more effective than the solvent hydrogen bond acceptor capacity (HBA) in directing to a negative solvatochromic behavior. The observed blue shift also suggests that the ground state has a better stabilization relative to the electronic excited state with increasing solvent polarity. Thus, molecules in the ground state possessed higher dipolar properties compared to molecules in the excited state [41].

A comparison has been made between the two solvent scales approach in Table 4 and it is observed that the use of Kamlet-Taft gives a better regression than that of Dimroth-Reicherdt's solvent scales. This outcome may be attributed to the functionality of various independent interactions in the Kamlet-Taft equation that are useful to characterize implicitly between solvent and solute ground, transition and surrounding [42].

Table 3 Solvatochromic parameters for P1–P3 using Kamlet-Taft approach

Polymers	$v_o \times 10^3$ (cm^{-1})	$s \times 10^3$ (cm^{-1})	$a \times 10^3$ (cm^{-1})	$b \times 10^3$ (cm^{-1})	P_{π^*} (%)	P_α (%)	P_β (%)
P1	23.57	1.18	1.13	1.02	35.44	33.93	30.63
P2	21.49	3.97	2.33	2.09	47.32	27.78	24.91
P3	22.51	1.12	0.97	0.88	37.77	32.66	29.63

Table 4 Experimental and theoretical absorption maxima for P1–P3 in various solvents

Polymers	Solvents	Observed value		Calculated by Kamlet-Taft		Calculated by Dimroth-Reicherdt	
		$\lambda_{max\ (nm)}$	ν_{max} ($\times 10^3$ cm^{-1})	λ_{max} (nm)	ν_{max} ($\times 10^3$ cm^{-1})	λ_{max} (nm)	ν_{max} ($\times 10^3$ cm^{-1})
P1	$CHCl_3$	398	25.13	398	25.13	397	25.19
	THF	397	25.19	397	25.19	399	25.06
	Chlorobenzene	401	24.94	401	24.94	399	25.02
	DCM	395	25.32	395	25.32	395	25.30
P2	$CHCl_3$	403	24.81	403	24.81	400	24.97
	THF	401	24.94	401	24.94	405	24.70
	Chlorobenzene	409	24.45	409	24.45	406	24.61
	DCM	396	25.25	396	25.25	397	25.22
P3	$CHCl_3$	424	23.58	424	23.58	424	23.59
	THF	423	23.64	423	23.64	425	23.51
	Chlorobenzene	428	23.36	428	23.36	426	23.48
	DCM	423	23.64	423	23.64	423	23.66

Substituent Effect

Figure 4(a) shows absorption spectra of P1–P3 in THF with a concentration of 1×10^{-6} M in the wavelength range from 300 to 600 nm. The absorption spectra of all the studied polymers are very similar in shape because of their structural similarity. The broad absorption band in the investigated compounds was appeared in the range of 397–423 nm. These absorption bands may be assigned as π–π* transition involving π conjugation throughout the whole mesogenic unit [28]. The absorption maxima (λ_{max}) are red shifted from P1 to P3 with increasing strength of electron donating group on the benzothiazole unit in the mesogenic side chain which due to extended resonance system [43]. The same trend was also observed in $CHCl_3$, DCM and chlorobenzene (data not shown). These shifts of absorbance maxima (λ_{max}) may be ascribed to the presence of an electron-donor substituent in the benzothiazole moiety that increase the absorption coefficient, thus enhances the polarization of the molecules [44]. Song co-workers observed a similar result for their studied compounds [45].

Figure 4(b) depicts photoluminescence (PL) spectra of P1–P3 in THF solutions (1×10^{-6} M) when excited at 430 nm. The emission spectra of the polymers show similar pattern as observed in UV-vis spectra. This is due to the structural similarities in the mesogenic unit. The fluorescence emission maxima of polymers are in the range 461–482 nm which may be categorized as blue emission. Likewise, PL emission maxima also exhibited a significant bathochromic shift (red shift) due to highly polarizable π-conjugated structures [29]. The polarity of the solvent also affects the electron delocalization. In higher polarity solvent, the relaxation process from the excited state to a lower energy state is easily achieved due to the greater polarity of the former state than the latter, which results in a red shift of the emission peak [45].

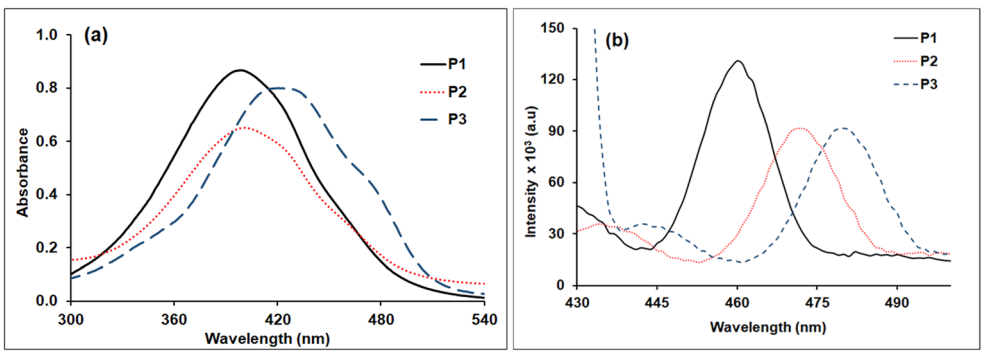

Fig. 4 (**a**) Absorption and (**b**) emission spectra P1–P3 dissolved in THF (1×10^{-6} M)

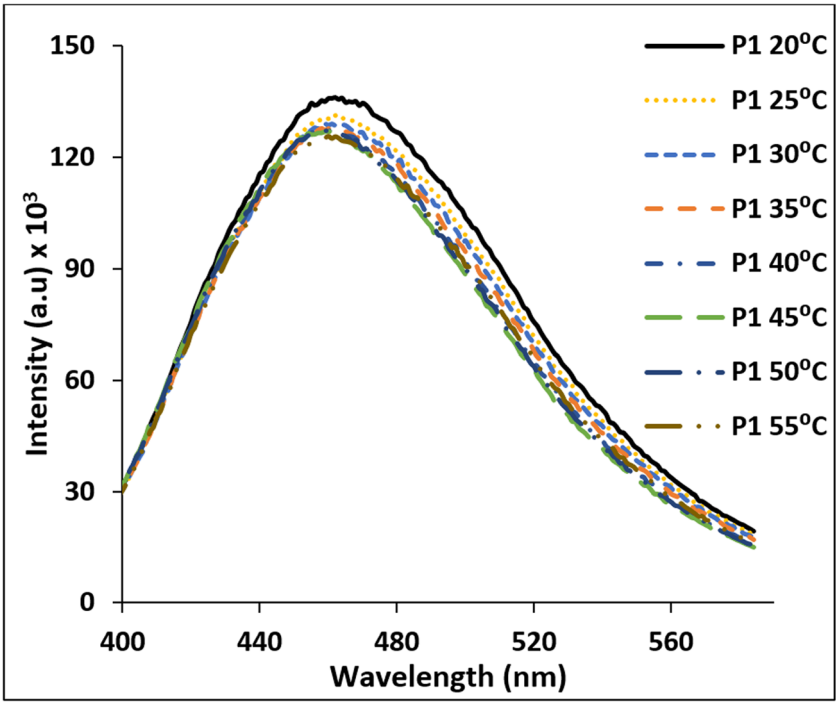

Fig. 5 Emission spectra of P1 dissolved in THF (1×10^{-6} M) at different temperatures

Temperature Effect

To study thermochromism of investigated polymers, the emission intensities and peak positions of P1–P3 were monitored in the temperature range from 20 °C to 55 °C. It is observed from Fig. 5 that the emission bands of P1 do not undergo any spectral shift upon increasing temperature. The result indicates that the temperature does not affect the excited states of conjugated polymer [34]. In addition, a virtually planar conformation in the molecular structure of these polymers also lead to the absence of any chromic effect [13].

In contrast, the intensity of emission bands for P1 exhibits a decreasing trend with the increasing temperature. A similar observation was reported in 1,6- and 1,7-naphthyridines compounds [46]. This result indicates that an increase in temperature could increase the molecular energy and charge delocalization, thus eventually weakens the intermolecular hydrogen bonding between the solutes and solvent [33]. Similar spectral behaviors were also observed for P2 and P3 (Supporting Information, Section C). Rageh [47] reported that this behavior could be correspond to an association process through solute–solvent interaction as the same phenomenon was also observed upon heating of azo cinnoline compounds. Conformational changes might also play a role in explaining the spectral shift in the temperature-dependent absorption spectra [13]. It can be concluded from above observations that an increase in the temperature could lead to an increase in the rate of dissociation as well as conformational changes. Thus, this will cause the concentration of complexed species as well as equilibrium of reaction to be decreased: HA + S↔ [AH—S], where HA and S is the solute and solvent respectively.

Conclusion

In this work, we have described the effect of solvent, substituent and temperature on absorption and emission spectra of azo-benzothiazole mesogen, containing SCLCPs, P1–P3 using UV-vis and fluorescence spectroscopies. Both electronic absorption and emission spectra of the investigated polymers did not show a regular variation in various solvent media. Correlations of UV-vis absorption frequencies with Dimroth-Reichardt's and Kamlet-Taft polarity scales revealed that the solvent hydrogen bond donor capacity (HBD) was more effective than the solvent hydrogen bond acceptor capacity (HBA) in directing to a negative solvatochromic behavior. Additionally, the absorption maxima of UV-vis spectra as well as fluorescence spectra were red shifted by the influence of electron donating substituents located at the sixth position on the benzothiazole core. The thermochromic effect of P1–P3 exhibited similar behavior, in which the emission intensities decreased without any spectral shift upon increasing temperature.

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