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ORIGINAL ARTICLE

1st Heterocyclic Update

Spectrophotometric and spectrofluorimetric studies of novel heterocyclic Schiff base dyes

Hadi M. Marwani [a,b], **Abdullah M. Asiri** [a,b], **Salman A. Khan** [a,*]

[a] *Department of Chemistry, Faculty of Science, King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Saudi Arabia*
[b] *Center of Excellence for Advanced Materials Research (CEAMR), King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Saudi Arabia*

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Abstract Novel heterocyclic Schiff base dyes were prepared by the reaction of salicylaldehyde/2-Hydroxy-1-naphthaldehyde with different heterocyclic amines. Results of the newly synthesized compounds established by FT-IR, ^{1}H-NMR, ^{13}C-NMR and GC–MS spectroscopic experiments were consistent with their chemical structures. Resulted heterocyclic Schiff base dyes were found to be pure from data obtained by the elemental analysis. In addition, spectrophotometric and spectrofluorimetric studies showed that these dyes were good absorbent and fluorescent. Fluorescence polarity study data revealed that some of these compounds were sensitive to the polarity of the microenvironment provided by different solvents. Time-based fluorescence steady-state measurements also showed that these heterocyclic Schiff base dyes have high photostability against photobleaching.

1. Introduction

Schiff base products are generally known as azomethine compounds due to the presence of azomethine bond (Asiri et al., 2010). They are derived from the condensation of amines with carbonyl compounds (Da-Silva et al., 2011). Schiff bases have a wide variety of applications in many fields, such as biological (Li and Liu, 2011), inorganic (Nair et al., 2012), analytical (Cimerman et al., 1997), organic (Praveen et al., 2008) and material chemistry (Brefuel et al., 2007). They are biologically active, used as antibacterial (Asiri and Khan, 2010), anti cancer (Singh et al., 2007), anti- inflammatory (Geronikaki et al., 2003) and anti-HIV (Shah and Desai, 2007). Schiff bases are very important class of organic compounds for inorganic chemistry because of their ability to form stable complex with different transition metals. These compounds are also used as intermediates for the formation of various heterocyclic compounds by a nucleophilic addition reaction (Sriram et al., 2006). Most Schiff bases synthesized by salicylaldehyde/2-Hydroxy-1-naphthaldehyde were chemically unstable and showed a tendency to be involved in various equilibria, like tautomeric interconversions, hydrolysis, or formation of ionized species (Nazıra et al., 2000; Bilgel et al., 2009). Therefore, a successful

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[*] Corresponding author. Tel.: +966 2 6952293; fax: +966 2 6952292.
E-mail address: sahmad_phd@yahoo.co.in (S.A. Khan).
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application of Schiff bases requires a careful study of their characteristics. In the last decades, the photophysical properties of organic compounds have much attention for their wide applications in material chemistry, such as, nonlinear optical properties (Sheikhshoaie and Fabian, 2006), photonic materials (Iwan and Sek 2011), devices (Niu et al., 2004), optical limiting (Kong et al., 2005), electrochemical sensing (Kaya et al., 2010), light-emitting devices (Sharbati et al., 2011), langmuir film (Jiao and Minghua Liu, 2006) and solar cell materials (Bhattacharjee et al., 2012). In accordance, this study was aimed to report the synthesis and photophysical properties of novel Schiff bases derived from salicylaldehyde/2-Hydroxy-1-naphthaldehyde and various heterocyclic amines.

2. Experimental

2.1. Instrumental methods

Melting points were recorded on a Thomas Hoover capillary melting apparatus without correction. FT-IR measurements were performed on KBr disks on a Nicolet Magna 520 FT-IR spectrometer. ^{1}H-NMR and ^{13}C-NMR spectroscopic experiments were recorded in $CDCl_3$ on Brucker DPX 600 and 125 MHz spectrometers, respectively, using tetramethyl silane (TMS) as an internal standard. Microanalyses were carried out using a Perkin Elmer 240B analyzer. The UV–Vis absorption measurements were acquired by use of a Perkin-Elmer UV–Vis scanning spectrophotometer. Absorption spectra were collected using a 10 mm quartz cuvet. Fluorescence measurements were performed using a Perkin-Elmer luminescence spectrofluorometer equipped with a 20-KW for 8 μs duration xenon lamp and gated photomultiplier tube (PMT) and red-sensitive R928 PMT detectors. All fluorescence measurements were collected at room temperature. The emission spectra were recorded in a 10 mm quartz fluorescence cuvette and excited at 375 nm for T1, 416 nm for T2, 350 nm for T3, 385 nm for T4, 360 nm for T5 and 400 nm for T6 with slit widths set for entrance and exit bandwidths of 3 and 3 nm on both excitation and emission monochromators, respectively. All fluorescence spectra were blank subtracted before proceeding in data analyses. Time-based steady-state fluorescence experiments were performed with excitation and emission bandpass set at 15 and 5 nm, respectively, in order to induce the photobleaching. The excitation and emission wavelengths were uniformly set at 380 and 470 nm, respectively, as the optimum conditions for all dyes included in this study in order to investigate differences in their photostability behaviors. The fluence level of the excitation source was open for a period of 30 min.

2.2. General method of the synthesis of Schiff bases

A mixture of salicylaldehyde/2-Hydroxy-1-naphthaldehyde (0.0058 mol) and the corresponding active heterocyclic amine (0.0058 mol) in anhydrous methanol (15 mL) were refluxed at 80 °C for 3 h with continuous stirring in the presence of a few drops of acetic acid. Progress of the reaction was monitored by TLC. After completion of the reaction, the solution was cooled. Thus, the heavy precipitate obtained was collected by filtration and purified by recrystallization from methanol and chloroform (T1–T6).

2.3. 2-[2-Hydroxy-benzylidene)-amino]-4,5,6,7-tetrahydro-benzo[b]thiophene-3-carbonitrile [T1]

Yield: 76.5%; M.p. 200–201 °C; GC–MS m/z (rel. int.%): 284 (78) $[M + 1]^+$; Anal. calc. for $C_{16}H_{14}N_2OS$: C, 68.06, H, 5.00, N, 9.92, Found: 67.98, H, 497, N. 9.89; FT-IR (KBr) ν_{max} cm^{-1}: 2945 (C–H), 1597 (C=C), 1557 (C=N), 1143 (C–N); ^{1}H NMR (600 MHz $CDCl_3$) δ: 12.01 (s, OH), 8.40 (s, 1H, CH olefinic), 7.42 (dd, H2, $J = 1.8$, 1.2 Hz), 7.49 (dd, H4, $J = 1.2$, 1.8 Hz), 7.36 (dd, H3, $J = 1.8$, 1.8 Hz), 6.96 (dd, H5, $J = 1.2$, 1.2 Hz), 2.72–1.82 (m, 8 H, $-CH_2$); ^{13}CNMR ($CDCl_3$) δ: 161.14, 160.82, 157.17, 135.13, 134.37, 133.00, 132.71, 119.60, 118.40, 117.64, 113.92, 106.53, 25.17, 24.25, 22.96, 21.86.

2.4. 2-[(2-Hydroxy-naphthalen-1-ylmethylene)-amino]-4,5,6,7-tetrahydro-benzo[b]thiophene-3-carbonitrile [T2]

Yield: 77.8%; M.p. 288–289 °C; GC–MS m/z (rel. int.%): 334 (73) $[M + 1]^+$; Anal. calc. for $C_{20}H_{16}N_2OS$: C, 72.26, H, 4.85, N, 8.43, Found: 72.23, H, 4.82, N, 8.39; FT-IR (KBr) ν_{max} cm^{-1}: 2940 (C–H), 1594 (C=C), 1552 (C=N), 1143 (C–N); ^{1}H-NMR (600 MHz, CDCl3) δ: 14.46 (s, OH), 9.41 (s, 1H, CH olefinic), 8.14 (d, H3, $J = 8.4$ Hz), 7.80 (d, H4, 7.8 Hz), 7.60 (dd, H5, $J = 1.2$, 1.8 Hz), 7.90 (dd, H6, $J = 1.8$, 1.2 Hz), 7.24 (d, H7, $J = 6.0$ Hz), 2.75–1.23 (m, 8H, $-CH_2$); ^{13}CNMR ($CDCl_3$) δ: 163.50, 157.63, 156.63, 135.14, 132.46, 136.38, 129.50, 124.07, 119.44, 119.44, 116.44, 114.21, 109.29, 105.44, 58.50, 25.22, 24.47, 23.02, 21.92, 18.00.

2.5. 2-[(3,4-Dimethyl-isoxazol-5-ylimino)-methyl]-phenol (T3)

Yield: 66.78%; M.p. 116 °C; GC–MS m/z (rel. int.%): 218 (56) $[M + 1]^+$; Anal. calc. for $C_{12}H_{12}N_2O_2$: C, 66.65, H, 5.59, N, 12.95, Found: 66.63, H, 5.54, N, 12.92;FT-IR (KBr) ν_{max} cm^{-1}: 2922 (C–H), 1595 (C=C), 1563 (C=N), 1156 (C–N); ^{1}H NMR (600 MHz $CDCl_3$) δ: 8.89 (s, 1H, CH olefinic), 7.42 (d, H3, $J = 1.8$ Hz), 7.44 (dd, H4, $J = 7.8$ Hz), 7.02 (dd, H5, $J = 7.8$ Hz), 6.97 (d, H6, $J = 1.2$ Hz), 2.25 (s, CH_3), 2.05 (s, CH_3); ^{13}CNMR ($CDCl_3$) δ: 162.87, 162.58, 161.21, 134.69, 134.51, 119.75, 118.84, 117.44, 109.32, 106.61, 10.74, 6.69.

2.6. 1-[(3, 4-Dimethyl-isoxazol-5-ylimino)-methyl]-naphthalen-2-ol (T4)

Yield: 74.65%; M.p. 160 °C; GC–MS m/z (rel. int.%): 268 (68) $[M + 1]^+$; Anal. calc. for $C_{16}H_{14}N_2O_2$: C, 72.17, H, 5.30, N, 10.52, Found: 72.15, H, 5.26, N, 10.49; FT-IR (KBr) ν_{max} cm^{-1}: 2933 (C–H), 1626 (C=C), 1585 (HC=N), 1123 (C–N); ^{1}H-NMR (600 MHz, CDCl3) δ: 14.46 (s, OH), 8.30 (d, $J = 12.7$ Hz, 1H, H-3), 7.99 (d, $J = 13.5$ Hz, 1H, H-4), 7.87 (d, $J = 11.8$ Hz, 1H, H-5), 7.70 (dd, $J = 8.6$ Hz, 1H, H-6), 7.51 (dd, $J = 10.8$ Hz, 1H, H-7), 7.28 (d, $J = 13.6$ Hz, 1H, H-8), 7.34 (s, 1H, CH olefinic), 2.36 (s, CH_3), 2.15 (s, CH_3); ^{13}C-NMR ($CDCl_3$) δ: 163.69, 162.76, 162.21, 158.27, 136.55, 132.85, 129.33, 128.28, 128.00, 124.19, 121.14, 119.78, 119.44, 105.57, 10.77, 6.71.

2.7. 2(Benzothiazol-2-yliminothyl)-phenol (T5)

Yield: 75.5% M.p. 258 °C; GC–MS m/z (rel. int.%): 256 (82) $[M + 1]^+$; Anal. calc. for $C_{10}H_{10}N_2OS$: C, 66.12, H, 3.96, N, 11.02, Found: 66.09, H, 3.93, N, 10.98; FT-IR (KBr) ν_{max} cm^{-1}: 2932(C–H), 1606(C=C), 1566 (C=N), 1156(C–N); ^{1}H NMR (600 MHz CDCl$_3$) δ: 12.26 (s, OH), 9.27 (s, CH olefinic), 7.98 (d, J = 7.8 Hz), 7.53 (dd, J = 1.2 Hz), 7.49 (dd, J = 1.2 Hz), 7.86 (d, J = 6.0 Hz); ^{13}C NMR (CDCl$_3$) δ: 207.71, 169.06, 167.43, 161.93, 151.41, 135.48, 134.60, 134.09, 126.74, 125.30, 125.01, 121.77, 119.84, 118.31, 117.68, 30.98.

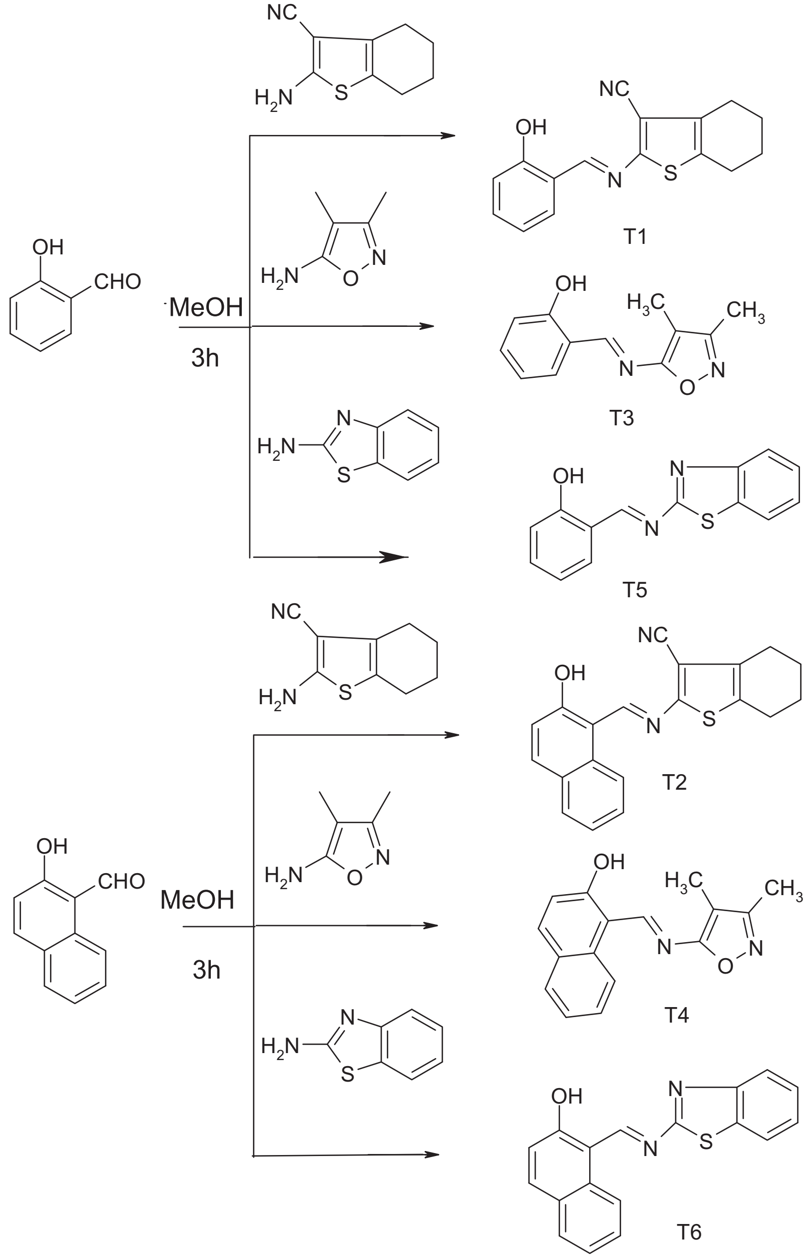
2.8. 1-(Benzothiazol-2-yliminomethyl)-naphthalen-2-ol (T6)

Yield: 72.56%; Mp. 214 C; GC–MS *m/z* (rel. int.%): 306 (86) [M + 1]$^+$; Anal. calc. for $C_{18}H_{12}N_2OS$: C, 71.03, H, 3.97, N, 9.20, Found: 70.98, H, 3.94, N, 9.18; FT-IR (KBr) ν_{max} cm^{-1}: 3018(C–H), 1591 (C=O), 1550 (C=N), 1142 (C–N); ^{1}H NMR (600 MHz CDCl$_3$) δ: 14.49 (s, OH), 10.14 (s, 1H, CH olefinic), 7.18–8.30 (d, H3, J = 8.4 Hz), 7.99 (d, H4, J = 8.4 Hz), 7.93 (d, H5, J = 9.0 Hz), 7.77 (dd, H6, J = 7.2 Hz), 7.49 (dd, H7, J = 7.2 Hz), 7.19 (d, H8, J = 9.0), 8.22–7.18 (4H, m, CH aromatic); ^{13}CNMR (CDCl$_3$) δ: 168.43, 166.73, 161.14, 151.54, 138.07, 134.35, 133.07, 129.46, 128.69, 127.91, 126.70, 125.04, 124.35, 122.73, 121.73, 120.13, 119.85, 109.85, 109.46, 30.98.

3. Results and discussion

3.1. Characterization

Schiff base derivatives **(T1–T6)** were prepared by the reaction of salicylaldehyde/2-Hydroxy-1-naphthaldehyde with different heterocyclic amines, previously described in the literature (Asiri and Khan, 2010). The synthetic route is outlined in Scheme 1. FT-IR spectra of Schiff bases (**T1–T6**) showed



Scheme 1 Synthetic route of the compounds T1–T6.

absorption bands at 2922–3014 cm^{-1} for aromatic C–H and at 1550–1566 cm^{-1} for the azomethine group (–CH=N–). The absence of the absorption band at 1700–1750 cm^{-1} also confirms the conversion of –CHO group to –CH=N– group. The ^1H NMR spectra of Schiff bases, as presented in the experimental section, showed peaks of methyl, olefinic (–N=CH–) and hydroxyl proton as singlets. The ^1H NMR spectra of dyes (**T1–T6**) showed sharp singlet at δ 7.34–10.14, confirming the presence of azomethine (–CH=N–) proton. The sharp singlet located at 12.01–14.49 indicated the presence of the hydroxyl group. The appearance of multiplets at δ 6.96–8.22 was due to aromatic protons.

Moreover, ^{13}C-NMR spectra showed the signals in the range of δ 134.25–136.55 ppm due to azomethine carbon, detailed studies of the ^{13}C-NMR spectra are given in the experimental section. Chemical structures of dyes (**T1–T6**) were further confirmed by the GC mass spectrum, all compounds showed the molecular ion peaks. A peak appeared at *m/z* 284 (M + 1, 78%), for dye **T1** having molecular formula ($C_{16}H_{14}N_2O_3$). FT-IR, ^1H NMR, ^{13}C NMR spectral data and elemental analysis results were also in agreement with the chemical structures of the dyes synthesized in this study. In addition, synthesized dyes (**T1–T6)** were found to be pure from data obtained by the elemental analysis, previously given in the experimental section.

3.2. Spectroscopic evaluation of dyes

3.2.1. Spectrophotometric study

UV–Vis absorption spectra of dyes were recorded under equivalent/similar experimental conditions (Fig. 1). Two characteristic broad bands in the UV–Vis absorption spectra of all dyes were observed, as illustrated in Fig. 1. In addition, the absorption spectrum of each dye included in this study exhibits a well defined maximum absorption wavelength due to the distinctive structural composition of these compounds.

3.2.2. Steady-state spectrofluorimetric polarity study

In general, high background fluorescence and similar spectral profiles were observed in the emission spectra of all compounds (Fig. 2). In this study, 10 μM standard solutions of each dye were prepared individually in different solvents (CHCl$_3$, EtOH, ACN, and DMF). According to the polarity index, DMSO is the most polar, while chloroform is the least polar (Snyder, 1978). The effect of polarity of different solvents in the emission spectra of T1 up to T6 was investigated. Minimal to no polarity effect of all solvents was observed in emission spectra of T1, T2, T3 and T5. However, it is of interest to note that polarity microenvironments of both T4 and T6 are affected by different solvents (Fig. 2). A red shift can be clearly observed in T4 and T6 emission spectra with an increase in the solvent polarity. A close inspection of Fig. 2 indicated that this red shift is more pronounced with fluorescence spectra of T6 (Fig. 2a) than those with T4 (Fig. 2a), particularly in the presence of DMF. This can be attributed to the distinctive structural composition of these two dyes. In fact, shifts in emission bands caused by a change in solvent polarity are called solvatochromic shifts and are experimental evidence of changes in solvation energy. The higher the polarity of the solvent, the lower is the energy of the relaxed state and the larger is the red-shift of the emission spectrum. The observed red (bathochromic) shift with increasing solvent polarity relates to a positive solvatochromism and indicates that a relaxed intramolecular charge transfer state is reached. Thus, the obtained results suggested that T6 is more sensitive than T4 to the polarity of the microenvironment provided by these solvents.

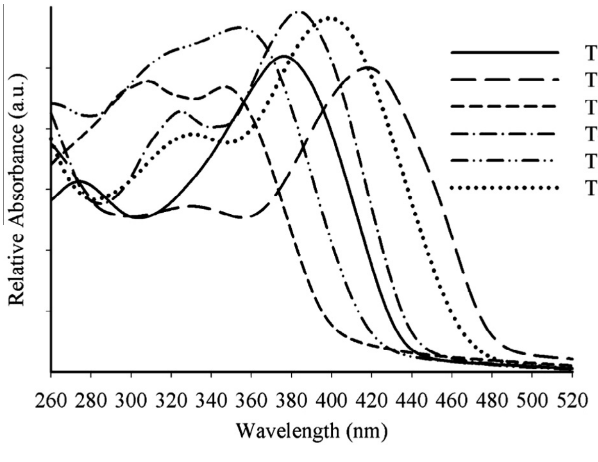


Figure 1 UV–Vis absorption spectra of 10 μM T1–T6 in ACN.

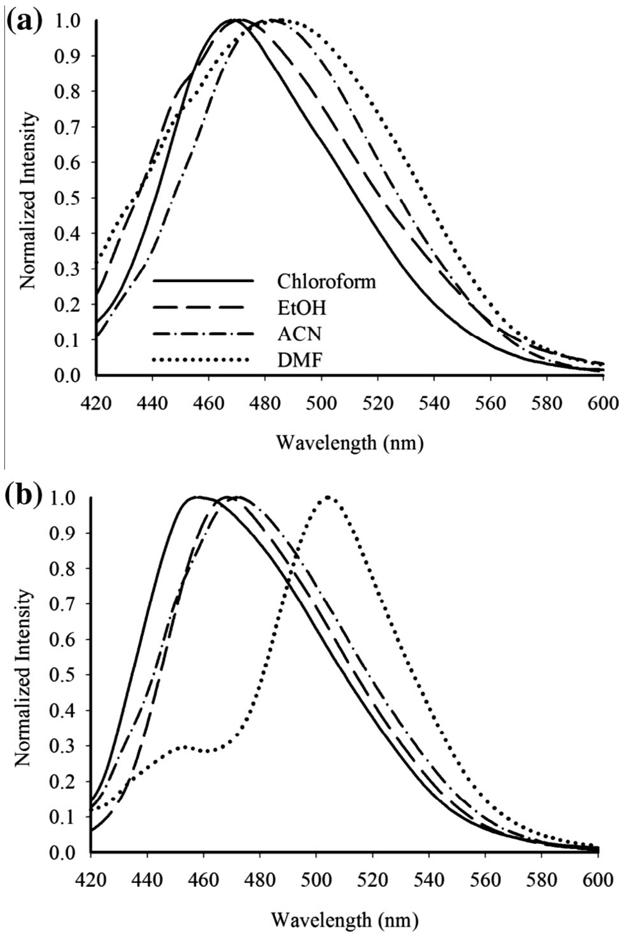


Figure 2 Normalized fluorescence emission spectra of 10 μM (a) T4 excited at 385 nm and (b) T6 excited at 400 nm in different solvents (CHCl$_3$, EtOH, ACN, and DMF).

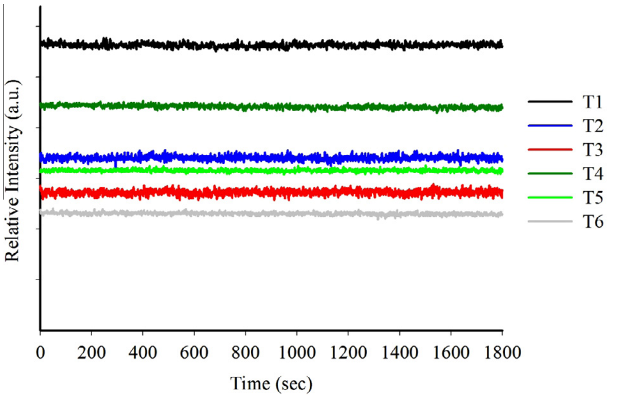


Figure 3 Time-based fluorescence steady-state measurements of 10 μM T1–T6 in ACN, excited at 380 nm and monitored at 470 nm with excitation and emission bandpass set at 15 and 5 nm, respectively.

3.2.3. Photostability study

Time-based steady-state fluorescence measurements were performed for all dyes in order to investigate their photostability against photobleaching. The excitation and emission bandpass set was at 15 and 5 nm, respectively, and all dyes have been exposed to the maximum amount of radiation for 30 min for induction of photobleaching. In addition, fluorescence measurements of all dyes were carried out on air-saturated samples for investigation of their photostability against photobleaching. Fig. 3 clearly shows the behavior of these dyes in terms of their susceptibility to photobleaching over a 30 min time span. It can be noted that there is minimal to no loss in fluorescence intensities of all dyes included in this study with an increase in the exposure time. The decrease in fluorescence intensities or % photobleaching of all dyes was also calculated from difference between the area under the emission peak of each dye before and after photobleaching (Fig. 4). The calculated % photobleaching of T1, T2, T3, T4, T5 and T6 was found to be 1.58%, 1.41%, 0.96%, 1.73%, 1.11% and 1.82%. Generally, information about the photostability relationship to the structure is so limited that the conclusion remains open. Based on the photostability study, it can be clearly noticed that T3 is the most photostable, while T6 is the least photostable against photobleaching.

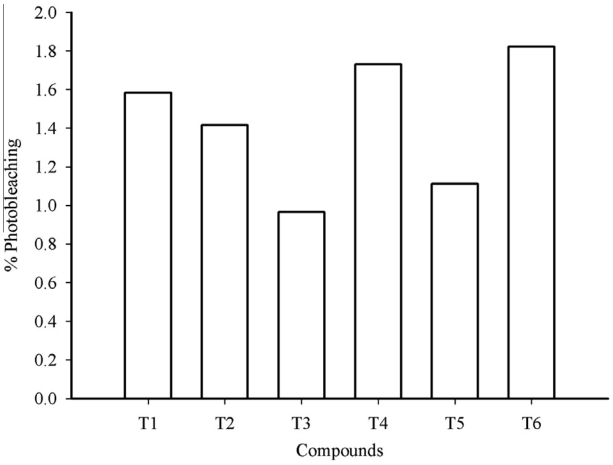


Figure 4 Calculated % photobleaching of T1–T6 in ACN based on time-based fluorescence steady-state experiments.

4. Conclusions

This study reported the preparation and spectroscopic properties of novel dyes. It was found that these dyes exhibited both significant absorption and high background florescence. Further, results of the photostability study suggested that these compounds had high photostability in terms of their susceptibility to photobleaching. Thus, these dyes can be used in gas chromatography or in high temperature reactions. Fluorescence polarity study data of the dyes revealed that both T4 and T6 were sensitive to the polarity of the microenvironment provided by different solvents. Moreover, solvatochromic shifts were observed in emission spectra of these dyes upon changing solvent polarities, indicating that these compounds can be used as solvatochromic probes. Finally, the preparation and spectroscopic evaluation of these dyes may show a considerable promise in a range of different applications, including analytical, biological and environmental.

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