Research Article

Synthesis of 2-(1-Benzofuran-2-yl)-4-(1,3-benzoxazol-2-yl/1,3-benzothiazol-2-yl) Quinolines as Blue Green Fluorescent Probes

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A series of novel 2-(1-benzofuran-2-yl)-4-(1,3 benzoxazol-2-yl/1,3-benzothiazol-2-yl) quinoline derivatives **4(a-d)** were synthesized in one step by the reaction of 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acids **3(a-b)** with o-aminophenol and o-amino thiophenol, respectively, using polyphosphoric acid (PPA) as a cyclizing agent. The fluorescent properties of newly synthesized compounds were investigated in three different organic solvents like chloroform (CHCl₃), tetrahydrofuran (THF), and dimethyl sulfoxide (DMSO). The photophysical constants such as quantum yield and stokes shift were determined. From the results of fluorescence study, it is evident that all synthesized compounds are fluorescent in solution. Compound **4a** emitted green light (490.4 nm, 518.2 nm, and 522.4 nm) with high quantum yield in all the three solvents, while compounds **4b**, **4c**, and **4d** emitted green light (512 nm, 499 nm, 510 nm) only in polar solvent DMSO. All fluorescent probes exhibited a bathochromic shift on increase in polarity of the solvent.

1. Introduction

There is an ever-increasing interest in the development of efficient photoluminescent materials especially those which emit light in the blue region of the visible spectrum. These materials play a vital role in optoelectronic devices such as tunable lasers and amplifiers, optical fibers, switches, or modulators with diverse applications in optical communications, photonics, medicines, optical spectroscopy, and organic electroluminescent diode (OLED) [1].

Among the various nitrogen-containing heterocyclic compounds, quinolines occur predominately in nature appreciable to their stability and ease of generation. They exhibit pronounced biological activities [2], and many derivatives have been reported to possess fluorescent property, having wide application as organic electroluminescent devices [3, 4], biosensors [5, 6], detection of metal ions [7–9], and so forth. On the other hand, benzofuran chromophore

with high photoluminescence (PL) and quantum efficiencies [10] found wide application as organic electroluminescent devices [11–13] and chemosensors [14, 15].

The present work describes the synthesis and characterization of six new dyes. Initially the two derivatives of 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acid **3(a-b)** were synthesized, and it was found that they emit light in the blue region of the spectrum. Further it was planned to modify the carboxylic acid functionality by electron donor benzoxazole and benzothiazole ring system. Therefore, the present investigation involves the synthesis and fluorescent studies of new 2-(1-benzofuran-2-yl)-4-(1,3-benzoxazol-2-yl/1,3-benzothiazol-2-yl) quinoline derivatives **4(a-d)** with a motive of getting possible application as blue OLE materials. Here we selected an unique combination of three different fused heterocyclic ring systems, where quinoline ring acts as core moiety, benzofuran nucleus substituted at second position acts as chromophore, and 3-benzoxazol-2-yl

(I) Aq. KOH/EtOH, reflux

(II) o-aminophenol/o-aminothiophenol/PPA

SCHEME 1

/benzothiazol-2-yl rings substituted on the fourth position of quinoline nucleus act as an electron acceptor system.

2. Result and Discussion

2.1. Chemistry. The photoluminescence properties of 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acids (**3a-b**) have prompted us to replace the carboxyl group with fused heterocyclic ring system like benzoxazole **4(a-b)** and benzothiazole **4(c-d)**, and to study the effect of alteration on the photoluminescence properties. The intermediate compounds **3(a-b)** were prepared by reported method [16], and structures were confirmed by spectral data. The title compounds 2-(1-benzofuran-2-yl)-4-(1, 3-benzoxazol-2-yl/ 1,3-benzothiazol-2-yl) quinolines (**4a-d**) were synthesized by reacting 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acids (**3a-b**) with o-aminophenol and o-amino thiophenol, respectively, using polyphosphoric acid as cyclizing agent. The structures of newly synthesized compounds were confirmed by IR, ^1H NMR, ^{13}C NMR, mass spectral data, and elemental analyses. The schematic representation of reaction has been shown in Scheme 1.

2.2. Fluorimetric Properties. In order to perform the spectral characterization of the new dyes, absorption and emission spectra of compounds **3(a-b)** were recorded in two different solvents, namely, THF and DMSO (as these two compounds are insoluble in chloroform), and absorption and emission spectra of compounds **4(a-d)** were recorded in CHCl_3 , THF, and DMSO. The absorption spectra have been shown in Figures 1(a)–1(c), and fluorescence spectra

in Figures 2(a)–2(f) and the relevant data is tabulated in Table 1. All fluorescent probes exhibited a bathochromic shift of the emission peak with decreased intensity of emitted light on raising the polarity of solvents, and a related increase of the Stokes shift and decrease in quantum yield are observed. Fluorescent probes **3a** and **3b** emitted light in the blue region of the spectrum, and 2-(1-benzofuran-2-yl)-6-chloroquinoline-4-carboxylic acid (**3b**) showed bathochromic shift by 11.5 nm when compared to 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acid **3a**. This may be due to the presence of electron releasing chloro substitution on the 6th position of quinoline ring system in compound **3b**, which acts as a chromophore. On replacing carboxylic group of 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acid with benzoxazole **4(a-b)** and benzothiazole **4(c-d)**, we found bathochromic shift in emission peaks and this may be attributed to the electron accepting tendency of benzoxazole and benzothiazole ring system, which decreases the energy gap between LUMO (π^*) and HOMO (π) leading to shift towards longer wavelength than compared to carboxylic acid. On increasing the solvent polarity, all compounds exhibited shift towards longer wavelength. The emission peaks are at lower energy or longer wavelength than compared to absorption peak; this loss of energy is due to a variety of dynamic processes, which occur following light absorption. The fluorophore is typically excited to the first singlet state (S_1), usually to an excited vibrational level within S_1 . The excess vibrational energy is rapidly lost to the solvent. If the fluorophore is excited to the second singlet state (S_2), it rapidly decays to the S_1 state in 10^{-12} s due to internal conversion. Solvent effects shift the emission to still lower energy owing to stabilization of the excited state by the polar solvent

FIGURE 1: Absorption spectra of compounds 3(a-b) and 4(a-d) in different solvents; 1(a) chloroform, 1(b) THF, and 1(c) DMSO.

molecules. Typically, the fluorophore has a larger dipole moment in the excited state than in the ground state. Following excitation, the solvent dipoles can reorient or relax at that state, which lowers the energy of the excited state. As the solvent polarity is increased, this effect becomes larger, resulting in emission at lower energies or longer wavelengths [16].

From the absorption data (Table 1), it is evident that the absorption wavelengths are slightly varied or no variations are observed on increasing polarity of the solvents. This may be because absorption of light occurs in about 10^{-15} s, a time that is too short for motion of the fluorophore or solvent. Absorption spectra are not affected by the decrease in the excited-state energy, which occurs after absorption has occurred [16].

Among the tested compounds, compound **4a** emitted light in the green region in all three solvents CHCl_3 , THF, and DMSO at wavelength of 490.4 nm, 518.2 nm, and 522.4 nm, respectively. Compound **4b**, **4c**, and **4d** emitted light in the green region only in polar solvent DMSO.

3. Experimental

All the chemicals used were of analytical grade. Melting points were determined in open capillary tubes and are uncorrected. Purity of the compounds was checked by TLC on silica gel. The IR spectra were recorded on Nicolet-Impact-410 FT-IR spectrometer, using KBr pellets. ^1H NMR spectra were recorded on a Bruker Supercon FT NMR (400 MHz)

FIGURE 2: Emission spectra of compounds 3(a-b) and 4(a-d) in different solvents.

TABLE 1: Photoluminescence properties of compound (3a-b) and (4a-d).

Compound	Solvent	Absorption $\lambda_{\max}$ nm	Emission $\lambda_{\max}$ nm	Molar absorptivity $\text{Mol}^{-1} \text{cm}^{-1}$	Stokes shift nm	Quantum yield Φ_F
3a	THF	418.0	451.6	223.6×10^4	33.6	0.183
	DMSO	423.2	483.8	153.9×10^4	60.6	0.165
3b	THF	430.4	462.8	305.8×10^4	32.4	0.186
	DMSO	430.4	491.8	197.3×10^4	61.4	0.181
4a	Chloroform	441.6	490.4	164.7×10^4	48.8	0.247
	THF	446.0	518.2	201.0×10^4	72	0.243
	DMSO	446.4	522.4	127.9×10^4	76	0.228
4b	Chloroform	432.0	485.8	228.4×10^4	53.8	0.184
	THF	432.2	470.2	216.4×10^4	38	0.167
	DMSO	442.0	512.0	713.2×10^4	70	0.132
4c	Chloroform	427.0	465.4	79.4×10^4	38.4	0.206
	THF	426.8	468.2	62.5×10^4	41.4	0.205
	DMSO	430.0	499.2	181.4×10^4	69.2	0.198
4d	Chloroform	427.0	468.2	76.1×10^4	41.2	0.143
	THF	426.8	478.0	97.1×10^4	51.2	0.136
	DMSO	423.6	510.6	69.2×10^4	87	0.134

spectrometer in CDCl_3 or $\text{DMSO}-d_6$ using TMS as an internal standard. The chemical shifts are expressed in δ units. Mass spectra were recorded on a JEOL SX 102/DA-6000 (10 kV) FAB mass spectrometer.

3.1. Synthesis of 2-(1-Benzofuran-2-yl) Quinoline-4-carboxylic Acid (3a-b)

3.1.1. General Procedure. The compounds **3a-b** were synthesized by the Pfizinger method [17], with the slight modification in procedure. After completion of the reaction, the reaction mixture was cooled to room temperature, diluted with cold water and extracted using ethyl acetate to suspend the impurities in organic layer. The aqueous layer was acidified with dilute hydrochloric acid and the resulting yellow solid mass was filtered and dried to get pure compound.

3.1.2. 2-(1-Benzofuran-2-yl) Quinoline-4-carboxylic Acid (3a). Yield, 88%, m.p. 245–248°C, (literature 248–50°C) [17], IR (KBr) (ν , cm^{-1}), 3426.9 (OH), 1702.1 (C=O), 1599, 1510, 1246; ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): 14.15 (s, 1H, COOH), 8.70–8.72 (d, J = 8.4, 1H Ar-H), 8.48 (s, 1H quinoline 3C-H), 8.18–8.20 (d, J = 8.4, 1H Ar-H), 8.014–8.018 (d, J = 16, 1H, Ar-H), 7.78–7.82 (q, 2H, Ar-H), 7.72–7.77 (t, 1H, Ar-H), 7.45–7.49 (t, 1H, Ar-H), 7.34–7.38 (t, 1H, Ar-H). MS: m/z = 290.1 (M + 1), 291.1 (M + 2).

3.1.3. 2-(1-Benzofuran-2-yl)-6-chloroquinoline-4-carboxylic Acid (3b). Yield, 76%, m.p. 283–285°C, IR (KBr) (ν , cm^{-1}) 3436 (OH), 1712 (C=O), 1570, 1514, 720, 640. ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): 13.85 (s, 1H, COOH) 8.80–8.82 (d, J = 8, 1H Ar-H) 8.55 (s, 1H quinoline 3C-H), 8.17–8.20 (d, J = 12, 1H Ar-H), 7.94 (s, 1H, furan-H), 7.89–7.91 (d, J = 8, 1H, Ar-H), 7.77–7.81 (q, 2H, Ar-H), 7.44–7.49 (t, 1H,

Ar-H), 7.34–7.37 (t, 1H, Ar-H). MS: m/z = 324.0 (M + 1). Anal. Calc. (in %) for $\text{C}_{18}\text{H}_{10}\text{NO}_3$: C, 67.05; H, 2.48; N, 4.32. Found: C, 67.25; H, 2.68; N, 4.43.

3.2. Synthesis of 2-(1-Benzofuran-2-yl)-4-(1,3-benzoxazol-2-yl)/1,3-benzothiazol-2-yl) Quinoline Derivatives 4(a-d)

3.2.1. General Procedure. A mixture of 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acid (**3a-b**) (0.5 mmol) and o-amino phenol/o-amino thiophenol was dissolved in 15 gm of polyphosphoric acid. The reaction mixture was heated with stirring at about 120–130°C for 8 hrs. After completion of the reaction, the reaction mixture was cooled, poured on to crushed ice, and neutralized using aqueous NaOH solution. The resulting solid was dried and purified by extracting using petroleum ether.

3.2.2. 2-(1-Benzofuran-2-yl)-4-(1, 3-benzoxazol-2-yl) Quinoline (4a). Yield, 66%, m.p. 162–164°C, ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): 9.45–9.49 (d, J = 16, 1H), 8.89 (s, 1H), 8.21–8.29 (t, J = 32, 1H), 7.958–8.009 (m, 4H), 7.831–7.899 (m, 3H), 7.602–7.644 (m, 2H), 7.469–7.581 (m, 1H), 7.361–7.402 (q, 1H), ^{13}C NMR (CDCl_3 , 100.5 MHz): 102.4 (1C), 103.2 (1C), 110.6 (1C), 111.5(1C), 119.1 (1C), 120.0 (1C), 120.9 (1C), 122.9 (1C), 123.8 (2C), 124.9 (1C), 125.1 (1C), 126.9 (1C), 128.3 (1C), 128.9(1C), 130.1 (1C), 142.2 (1C), 144.9(1C), 147.5 (1C), 149.9 (1C), 150.0 (1C), 155.3 (1C), 158.1 (1C), 162.7 (1C), MS: m/z = 363.1 (M + 1), 364.2 (M + 2). Anal.Calc. (in %) for $\text{C}_{24}\text{H}_{14}\text{N}_2\text{O}_2$: C, 79.86; H, 3.86; N, 7.72. Found: C, 79.40; H, 3.95; N, 7.62.

3.2.3. 2-(1-Benzofuran-2-yl)-4-(1, 3-benzoxazol-2-yl)-6-chloroquinoline (4b). Yield, 58%, m.p. 145–148°C, ^1H NMR ($\text{DMSO}-d_6$, 400 MHz): 9.599 (s, 1H), 8.898 (s, 1H),

8.201–8.223 (*d*, *J* = 8.8, 1H), 7.993–7.995 (*d*, *J* = 0.8, 1H), 7.684–7.977 (*m*, 5H), 7.401–7.484 (*m*, 2H), 7.324–7.343 (*t*, 1H), 7.25–7.268 (*m*, 1H), ¹³CNMR (CDCl₃, 100.5 MHz): 102.4 (1C), 103.2 (1C), 110.6 (1C), 111.5 (1C), 119.1 (1C), 120.0 (1C), 120.9 (1C), 122.9 (1C), 123.8 (2C), 124.9 (1C), 125.1 (1C), 125.8 (1C), 130.1 (1C), 131.2 (1C), 132.6 (1C), 142.2 (1C), 144.9 (1C), 147.5 (1C), 149.9 (1C), 150.0 (1C), 155.3 (1C), 158.1 (1C), 162.7 (1C). MS: *m/z* = 397.8 (*M* + 1), 399.8 (*M* + 2). Anal. Calc. (in %) for C₂₄H₁₃ClN₂O₂: C, 72.57; H, 3.25; N, 7.02. Found: C, 72.23; H, 3.85; N, 6.60.

3.2.4. 2-(1-Benzofuran-2-yl)-4-(1, 3-benzothiazol-2-yl) Quinoline (4c). Yield, 63%, m.p. 178–180°C, ¹H NMR (DMSO-*d*₆, 400 MHz): 8.90–8.98 (*d*, *J* = 32, 1H), 8.56(*s*, 1H), 8.21–8.42 (*m*, *J* = 84, 3H), 8.02 (*s*, 1H), 7.939–7.963 (*q*, 1H), 7.787–7.844 (*m*, 3H), 7.626–7.728 (*m*, 2H), 7.465–7.504 (*q*, 1H), 7.361–7.463 (*q*, 1H), ¹³CNMR (CDCl₃, 100.5 MHz): 102.4 (1C), 103.2 (1C), 111.5 (1C), 119.1 (1C), 120.0 (1C), 120.9 (1C), 121.8 (1C), 122.9 (1C), 123.8 (1C), 125.1 (1C), 125.6 (1C), 126.0 (1C), 126.9 (1C), 128.3 (1C), 128.9 (1C), 130.1 (1C), 135.5 (1C), 144.9 (1C), 147.5 (1C), 149.9 (1C), 153.5 (1C), 155.3 (1C), 158.1 (1C), 162.7 (1C), MS: *m/z* = 379.4 (*M* + 1). Anal. Calc. (in %) for C₂₄H₁₄N₂OS: C, 76.16; H, 3.69; N, 7.39. Found: C, 76.03; H, 3.55; N, 7.30.

3.2.5. 2-(1-Benzofuran-2-yl)-4-(1, 3-benzothiazol-2-yl)-6-chloroquinoline (4d). Yield, 70%, m.p. 165–168°C, ¹H NMR (DMSO-*d*₆, 400 MHz): 9.16 (*s*, 1H), 8.78 (*s*, 1H), 8.201–8.223 (*d*, *J* = 8.8, 1H), 7.993–7.995 (*d*, *J* = 0.8, 1H), 7.686–7.979 (*m*, 5H), 7.401–7.484 (*m*, 2H), 7.324–7.343 (*t*, 1H), 7.25–7.268 (*m*, 1H), ¹³CNMR (CDCl₃, 100.5 MHz): 102.4 (1C), 103.2 (1C), 111.5 (1C), 119.1 (1C), 120.0 (1C), 120.7 (1C), 121.8, 122.9 (1C), 123.8 (1C), 125.1 (1C), 125.6 (1C), 125.9 (1C), 126.0, 130.5 (1C), 131.5 (1C), 132.6 (1C), 135.5, 144.9 (1C), 146.1 (1C), 149.9 (1C), 153.5, 155.3 (1C), 158.1 (1C), 162.7 (1C). MS: *m/z* = 413.9 (*M* + 1), 414.9 (*M* + 2). Anal. Calc. (in %) for C₂₄H₁₃ClN₂OS: C, 69.8; H, 3.14; N, 6.78. Found: C, 69.4; H, 3.22; N, 6.67.

3.3. Fluorescent Studies. The fluorescence spectra were recorded on F-7000 spectrofluorometer (Hitachi) with Xenon flash lamp. The spectra were recorded by preparing the solutions of test compounds at the concentration of 5 × 10⁻⁶ mole/mL in three different solvents CHCl₃, THF, and DMSO. The solutions of each sample were scanned after keeping it at room temperature for about 1 h. The emission and excitation wavelength data are recorded using glass cuvettes. The emission quantum yields were determined at λ_{exc} corresponding to the maximum of the absorption band (λ_{max}) of quinine sulphate in 0.1 M H₂SO₄, which was used as a fluorimetric standard (Φ_F = 0.54).

4. Conclusion

In conclusion, we have demonstrated a convenient and efficient method for the synthesis of an important class of 2-(1-benzofuran-2-yl)-4-(1,3-benzoxazol-2-yl/ benzothiazol-2-yl) quinolines (**4a–d**) by reacting 2-(1-benzofuran-2-yl) quinoline-4-carboxylic acids (**3a–b**) with *o*-aminophenol and *o*-aminothiophenol respectively using PPA as cyclizing agent. All synthesized compounds displayed good fluorescent properties.

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