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Synthesis and fluorescent properties of some benzothiazole derivatives synthesized *via* Suzuki cross coupling reaction

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Fifteen compounds containing benzothiazole have been synthesized successfully *via* Suzuki reaction of 2-(4-bromophenyl)benzothiazole **1** and phenylboronic acid derivatives in high yield. All samples display fluorescence emissions at wavelengths in the range of 380 to 450 nm under the excitation light of 330 nm. Compounds **2d**, **2e**, **2h** and **2k** exhibit quite good fluorescent property.

Keywords: Benzothiazole, Suzuki cross coupling reaction, Fluorescence, Fluorescent property

Benzothiazole derivatives are a group of substances that are interested and researched by many scientists because they exhibit many important pharmacological effects such as anti-inflammatory[1], antibacterial[2], antiviral[3], antioxidant[4], immunomodulatory properties[5], anticancer[6,7] and especially, benzothiazole compounds have been reported to display selective inhibitory effects against monoamine oxidase[8-11] as well as anti-Alzheimer's disease[12] and convulsions[13].

Recently, the fluorescence properties of benzothiazole derivatives have been of special interest to many scientists. Specifically, the fluorescence properties of some 2-aryl- and 2-heteroaryl-benzothiazole were investigated in different solvents and *p*H (Ref. 14). Benzothiazole derivatives are used as fluorescent probes to detect β-amyloid and α-synuclein aggregates[15] and especially, benzothiazole derivatives were also used in the preparation of produce a white light emitting device[16].

Taking advantages of benzothiazole derivatives in interesting fluorescence properties and their potentially practical applications, we developed a process for synthesizing compounds containing benzothiazole *via* Suzuki reaction of 2-(4-bromophenyl)benzothiazole **1** with various phenylboronic acid derivatives with the goal of researching the fluorescence properties of the synthesized compounds.

Experimental Section

Palladium(II) chloride ($PdCl_2$) was purchased from Sigma-Aldrich; DMF was bought from Merck and used as received. Arylboronic acid were ordered on Aladdin.com from China. The 1D and 2D NMRs spectra were recorded on the Bruker Avance 500 NMR spectrometer in $CDCl_3$ at Institute of Chemistry, VAST. Mattson 4020 GALAXY Series FT-IR, LC-MSD-Trap-SL spectrometers were used to take IR and MS spectra. Progress of the reaction was monitored by thin-layer chromatography (TLC). The determination of melting points was carried out on a Gallenkamp melting-point apparatus in the opening in a capillary tube. The electronic absorption spectra were measured at the Department of Environment, Faculty of Chemistry, Hanoi National University of Education on the UV - Vis - NIR spectrometer; Model: UV-2600. Fluorescence spectra were measured on the Cary Eclipse Fluorescence Spectrophotometer of Varian at the Institute of Physics - Vietnam Academy of Science and Technology.

Fluorescence measurement

Electron absorption spectra of 10^{-3}M solution **2e** and **2k** in methanol were measured at RT. The fluorescence spectrum of the solution of **2b** - **2o** compounds at a concentration of 10^{-3}M was measured in methanol with an excitation wavelength of 330 nm. For the 5 samples **2d**, **2e**, **2g**, **2h** and **2k**, fluorescence spectra were further measured at concentrations of 10^{-4}M and 10^{-5}M.

Synthesis of 2-(4-bromophenyl)benzothiazole, 1

Synthesis procedure was followed with the protocol of Duong *et al.*[17,2]

General experimental procedure for the synthesis of benzothiazole derivatives, 2a-o

To a solution of K_2CO_3 (380 mg, 2.75 mmol), $PdCl_2$ (1.24 mg, 0.007 mmol), 2-phenylimidazole (2 mg, 0.014 mmol), 2-(4-bromophenyl) benzothiazole (**1**, 397 mg, 1.37 mmol) and aryl boronic acid (2.06 mmol) in anhydrous DMF (12 mL) was degassed with argon. Then the resulting mixture was stirred at 120℃ for 17h - 48h under argon atmosphere. The mixture was cooled and then poured over ice-water (250 mL) containing 1M HCl (2 mL) aqueous solution and extracted with $CHCl_3$ (5 × 20 mL). The combined organic layers were washed with brine (2 × 30 mL) and dried with Na_2SO_4. Removal of solvent followed by column chromatography purification (silica gel, hexane–$CHCl_3$) afforded the coupling product **2a-o** (Scheme 1).

Synthesis and structural determination of compounds **2a**, **2b**, **2e**, **2f**, and **2m** were reported in our previous paper[24].

2-(3',5'-Dimethyl-[1,1'-biphenyl]-4-yl)benzothiazole, 2c

Colourless needles. Yield 94%. m.p.190. 1-191.0℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.37 (9H, 3s, CH_3), 7.38 (1H, t, J = 7.5 Hz, CH), 7.48 (1H, t, J = 7.5 Hz, CH), 7.49 (2H, d, J = 7.5 Hz, *meta* CH), 7.60 (2H, d, J = 8.5 Hz, *ortho* CH), 7.71 (2H, d, J = 8.0 Hz, *meta* CH), 7.90 (1H, d, J = 8.0 Hz, CH), 8.08 (1H, d, J = 8.5 Hz, CH), 8.14 (2H, d, J = 8.0 Hz, *ortho* CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 31.3 $(CH_3)_3$-C), 34.6 (C-$(CH_3)_3$), 121.6, 123.1, 125.1, 125.9, 126.3, 126.7, 127.4, 128.0, 128.9, 132.2, 135.0, 137.1, 143.6, 151.1, 154.2 (aromatic and olefinic), 167.42 (S-C=N); ESI-MS: [M+H$^+$], *m/z* 344.0.

2-(2'-Methyl-[1,1'-biphenyl]-4-yl)benzothiazole, 2d

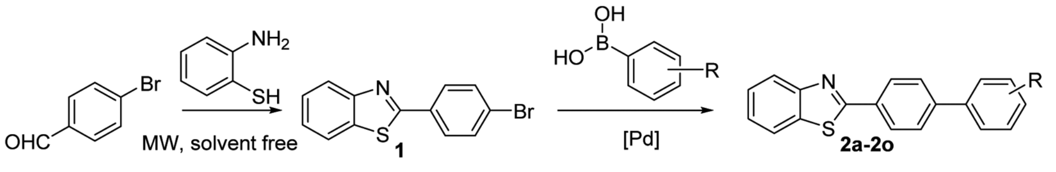
Colourless needles. Yield 87%. m.p. 123.5-124.1°C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.41 (3H, s, CH_3), 7.26-7.30 (4H, m, CH), 7.40 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.46 (2H, dd, J = 6.5 Hz and J = 1.5 Hz, meta CH), 7.50 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.91 (1H, d, J = 7.5 Hz, CH), 8.09 (1H, d, J = 8.0 Hz, CH), 8.14 (2H, dd, J = 6.5 Hz and J = 2.0 Hz, ortho CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 20.4 (CH_3), 121.6, 123.2, 125.2, 125.9, 126.4, 127.4, 127.7, 129.6, 129.9, 130.5, 132.0, 135.0, 135.3, 140.9, 144.9, 154.1, (aromatic and olefinic), 167.9 (S-C=N); ESI-MS: [M+H$^+$], m/z 301.9 [M+H$^+$].

2-(4'-(Trifluoromethyl)-[1,1'-biphenyl]-4-yl) benzothiazole, 2g

Colourless needles. Yield 82%. m.p. 206.5-207.0°C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.50 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.71 (2H, d, J = 8.5 Hz, meta CH), 7.73 (2H, d, J = 8.5 Hz, meta CH), 7.74 (2H, d, J = 8.5 Hz, ortho CH), 7.90 (1H, dd, J = 8.0 Hz and J = 0.5 Hz, CH), 8.09 (1H, d, J = 8.5 Hz, CH), 8.19 (2H, dd, J = 8.0 Hz and J = 1.5 Hz, ortho CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 121.6, 123.4, 125.4, 125.8, 125.8, 125.9, 126.5, 127.4, 127.8, 128.2, 133.5, 135.2, 142.2, 143.7. 154.2, (aromatic and olefinic), 167.31 (S-C=N); ESI-MS: [M+H-CF_3^+], m/z 288.1.

2-(3'-Ethoxy-[1,1'-biphenyl]-4-yl)benzothiazole, 2h

Colourless needles. Yield 90%. m.p, 142-143℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.45 (3H, s, CH_3-CH_2), 4.10 (2H, q, J = 2.0 Hz, OCH_2-CH_3), 6.92 (1H. ddd, J = 8.5 Hz and J_1 = 2.0 Hz and J_2 = 0.5 Hz, CH), 7.21 (1H, dq, J = 7.5 Hz and J = 0.5 Hz, CH), 7.27 (1H, t, J = 2.0 Hz, CH), 7.36 (1H, t, J = 8.0 Hz, CH), 7.39 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.49 (1H, td, J = 8.0 Hz and J = 1.0 Hz, CH), 7.70 (2H, dt, J = 8.5 Hz and J = 2.0 Hz, *meta* CH), 7.89 (1H, dd, J = 8.0 Hz and J = 0.5 Hz, CH), 8.08 (1H, d, J = 8.0 Hz, CH), 8.14 (2H, dt, J = 8.5 Hz and J = 2.0 Hz, *ortho* CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.9 (CH_3-CH_2), 63.5 (OCH_2-CH_3) 113.5, 113.8, 119.5, 121.6, 123.2, 125.2, 126.3, 127.6, 127.9, 129.9, 132.6,



Scheme 1 — Synthesis of coupling products **2a-o**

135.1, 141.5, 143.6, 154,2 (aromatic and olefinic), 159,47 (C-O aromatic), 167.69 (S-C=N); ESI-MS: [M+H$^+$], *m/z* 331.9.

2-(3'-Chloro-[1,1'-biphenyl]-4-yl)benzothiazole, 2i

Colourless needles. Yield 90%. m.p. 148.2-149.0℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.35 (1H, dt, *J* = 8.0 Hz and *J* = 2.0 Hz, CH), 7.37 (1H, t, *J* = 8.0 Hz, CH), 7.38 (1H, td, *J* = 8.0 Hz and *J* = 1.0 Hz, CH), 7.49 (1H, td, *J* = 7.5 Hz and *J* = 1.5 Hz, CH), 7.50 (1H, dt, *J* = 7.5 Hz and *J* = 1.5 Hz, CH), 7.62 (1H, d, *J* = 2.0 Hz, CH), 7.66 (2H, dt, *J* = 8.5 Hz and *J* = 2.0 Hz *meta* CH), 7.89 (1H, d, *J* = 8.0 Hz, CH), 8.08 (1H, d, *J* = 8.0 Hz, CH), 8.15 (2H, dt, *J* = 8.5 Hz and *J* = 2.0 Hz, *ortho* CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 121.6, 123.2, 125.2, 125.3, 126.4, 127.2, 127.6, 127.9, 128.1, 130.1, 133.0, 134.9, 135.0, 141.9, 142.2, 154.1 (aromatic and olefinic), 167.42 (S-C=N); ESI-MS: [M+H-Cl$^+$], *m/z* 288.1.

2-(4'-(Tert-butyl)-[1,1'-biphenyl]-4-yl)benzothiazole, 2j

Colourless needles. Yield 91%. m.p. 190.1-191.0℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.37 (9H, 3s, CH_3), 7.38 (1H, t, *J* = 7.5 Hz, CH), 7.48 (1H, t, *J* = 7.5 Hz, CH), 7.49 (2H, d, *J* = 7.5 Hz, *meta* CH), 7.60 (2H, d, *J* = 8.5 Hz, *ortho* CH), 7.71 (2H, d, *J* = 8.0 Hz, *meta* CH), 7.90 (1H, d, *J* = 8.0 Hz, CH), 8.08 (1H, d, *J* = 8.5 Hz, CH), 8.14 (2H, d, *J* = 8.0 Hz, *ortho* CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 31.3 ($(CH_3)_3$-C), 34.6 (C-$(CH_3)_3$), 121.6, 123.1, 125.1, 125.9, 126.3, 126.7, 127.4, 128.0, 128.9, 132.2, 135.0, 137.1, 143.6, 151.1, 154.2 (aromatic and olefinic), 167.42 (S-C=N); ESI-MS: [M+H$^+$], *m/z* 344.0.

2-(4'-Butyl-[1,1'-biphenyl]-4-yl)benzothiazole, 2k

Colourless needles. Yield 94%. m.p. 168.0-169.0℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95 (3H, t, *J* = 7.5 Hz, CH_3-CH_2), 1.39 (2H, m, CH_2-CH_3), 1.64 (2H, m, CH_2-CH_2-CH_3), 2.66 (2H, t, *J* = 7.5 Hz, CH_2-Ar), 7.27 (2H, d, *J* = 8.0 Hz, *meta* CH), 7.37 (1H, td, *J* = 7.5 Hz and *J* = 1.0 Hz, CH), 7.49 (1H, td, *J* = 8.0 Hz and *J* = 1.0 Hz, CH), 7.56 (1H, dt, *J* = 8.5 Hz and *J* = 2.0 Hz, *ortho* CH), 7.70 (2H, dt, *J* = 8.5 Hz and *J* = 2.0 Hz, *meta* CH), 7.89 (1H, dd, *J* = 8.0 Hz and *J* = 0.5 Hz, CH), 8.08 (1H, d, *J* = 8.0 Hz, CH), 8.14 (2H, dt, *J* = 8.5 Hz and *J* = 2.0 Hz, *ortho* CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 13.9 (CH_3-CH_2), 22.4 (CH_2-CH_3), 33.6 (CH_2-CH_2-CH_3), 35.3 (CH_2-Ar), 121.6, 123.1, 125.1, 126.3, 126.9, 127.4, 128.0, 129.0, 132.1, 135.0, 137.3, 142.9, 143.7, 154.1 (aromatic and olefinic), 167.8 (S-C=N); ESI-MS: [M+H$^+$], *m/z* 344.0.

2-(4'-Chloro-[1,1'-biphenyl]-4-yl)benzothiazole, 2l

Colourless needles. Yield 92%. m.p. 179.0-179.5°C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 (1H, td, *J* = 8.0 Hz and *J* = 1.0 Hz, CH), 7.43 (2H, dt, *J* = 8.5 Hz and *J* = 2.5 Hz, meta CH), 7.50 (1H, td, *J* = 7.5 Hz and *J* = 1.5 Hz, CH), 7.56 (2H, dt, *J* = 7.5 Hz and *J* = 2.5 Hz, ortho CH), 7.67 (2H, dt, *J* = 8.5 Hz and *J* = 1.5 Hz, meta CH), 7.90 (1H, dd, *J* = 8.0 Hz and *J* = 0.5 Hz, CH), 8.08 (1H, d, *J* = 7.0 Hz, CH), 8.15 (2H, dt, *J* = 8.0 Hz and *J* = 2.0 Hz, ortho CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 121.6, 123.2, 125.3, 126.4, 127.4, 128.1, 128.3, 129.1, 132.7, 134.1, 135.0, 138.5, 142.4, 154.1 (aromatic and olefinic), 167.49 (S-C=N); ESI-MS: [M+H$^+$], m/z 321.9.

2-(4-(Naphthalen-1-yl)phenyl)benzothiazole, 2n

Pale yellow solid. Yield 98%. m.p. 211.0-212.0°C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (1H, t, *J* = 7.5 Hz, CH), 7.45-7.48 (3H, m, CH), 7.51 (1H, td, *J* = 8.0 Hz and *J* = 1.0 Hz, CH), 7.55 (1H, t, *J* = 7.5 Hz, CH), 7.64 (2H, dd, *J* = 7.0 Hz and *J* = 1.5 Hz, meta CH), 7.90 (1H, d, *J* = 8.0 Hz, CH), 7.92-7.94 (3H, m, CH), 8.11 (1H, d, *J* = 8.0 Hz, CH), 8.22 (2H, dd, *J* = 6.5 Hz and *J* = 1.5 Hz, ortho CH); ^{13}C NMR (125 MHz, $CDCl_3$): δ 121.6, 123.2, 125.3, 125.3, 125.7, 125.9, 126.3, 126.4, 126.9,.127.1, 127.5, 128.2, 128.4, 128.9, 130.7, 131.3, 132.5, 133.8, 139.2, 143.7, 154.0 (aromatic and olefinic), 167.4 (S-C=N); ESI-MS: [M+H$^+$], m/z 337.9.

4-(Benzothiazol-2-yl)phenyl bis(cyclopentadienyl) iron, 2o

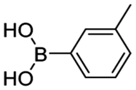
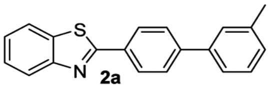
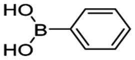
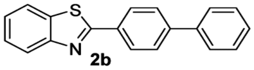
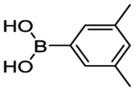
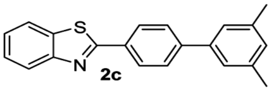
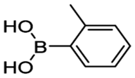
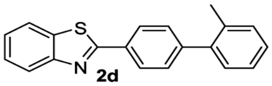
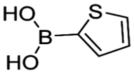
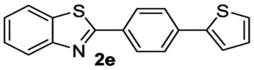
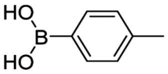
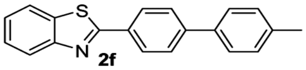
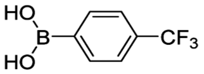
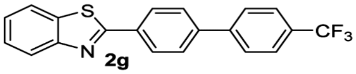
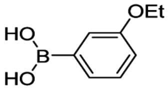
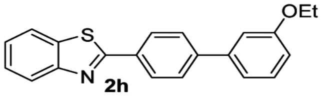
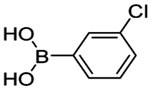
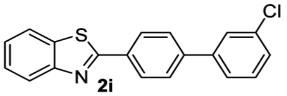
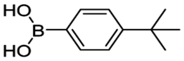
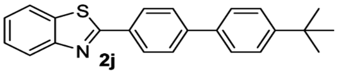
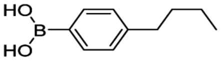
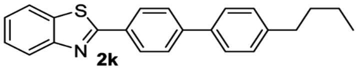
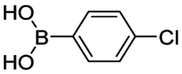
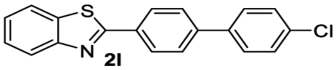
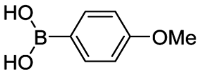
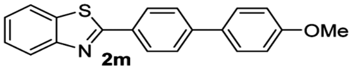
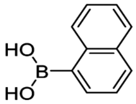
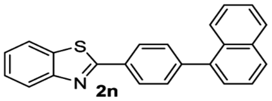
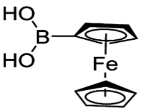
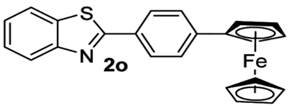
Brown solid. Yield 78%. m.p. 198.0-192.5℃. ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.06 s (2H, CH), 4.39 (1H, s, CH), 4.73 (1H, s, CH), 7.37 (1H, s, CH), 7.52 (1H, d, *J* = 38.5 Hz, CH), 7.89 (1H, d, *J* = 5.0 Hz, CH), 8.03 (1H, d, *J* = 38.5 Hz, CH); ^{13}C NMR: δ 66.90, 67.05, 69.86, 70.05, 121.61, 123.07, 125.02, 126.37, 127.63, 131.07, 134.99, 143.10, 154.31 (aromatic and olefinic), 168.09 (S-C=N); ESI-MS: [M+H$^+$], *m/z* 395.8.

Results and Discussion

Synthesis

Compound 2-(4-bromophenyl)benzothiazole **1** was synthesized by following the protocol of Hoan *et al.*[17,19] Suzuki reaction between 2-(4-bromophenyl)benzothiazole **1** and phenylboronic acid derivatives was carried to form 15 derivatives formed in high yield, up to 97% for 17-48 h (Table 1). Four compound **2**, **5**, **6** and **13** was reported by

Table 1 — Arylboronic acid derivatives and compounds containing benzothiazole

Entry	Arylboronic acid	Product	Time (h)	Yield (%)
1		 **2a**	26	97
2		 **2b**	21	95
3		 **2c**	28	94
4		 **2d**	27	87
5		 **2e**	18	95
6		 **2f**	25	84
7		 **2g**	17	82
8		 **2h**	32	90
9		 **2i**	23	90
10		 **2j**	35	91
11		 **2k**	37	94
12		 **2l**	20	92
13		 **2m**	30	96
14		 **2n**	45	98
15		 **2o**	48	78

Dhayalan *et al.*[18] It seems that the size of substituents of phenylboronic acid derivatives affects the time of completion of reactions. For example, compounds **2n** and **2o** have a very long reaction time, so it took about 45 - 48 h to finish.

Fluorescent properties

To establish the limitation of fluorescence spectroscopy of the series, compound **2e** and **2k** were selected to predict the fluorescence ability to measure electron absorption spectra.

Electron absorption spectra of **2e** and **2k** solutions in methanol, concentration 10^{-3}M was showed in Fig. 1.

There are 2 absorption peaks λ_{max} about 210 nm and 330 ÷ 340 nm with lg ε > 3 on UV-Vis absorption spectrum of **2e** and **2k** solutions at 10^{-3}M, there are 2 absorption peaks λ_{max} about 210 nm and 330 ÷ 340 nm with lg ε > 3. The resulting absorption fringes are due to the $\pi \rightarrow \pi^*$ or $n \rightarrow \pi^*$ transitions in **2e** and **2k**. Based on the results of electron absorption spectroscopy, the fluorescence spectrum of the **2b - 2o** solutions were measured at concentration of 10^{-3}M with an excitation wavelength of 330 nm. The fluorescence spectra of the solutions of **2b - 2o** compounds were showed in Fig. 2.

Under 330 nm excitation light, the solutions of **2b - 2o** compounds emit a wavelength at about 380 - 450 nm, corresponding to blue light and with varying fluorescence intensity depending on the structure of each compounds. Compounds have long conjugated chains by forming complexes with metals or bearing substituents causing ±I, +C, and ±H electron effects all increase the fluorescence intensity. As the results, compounds **2d**, **2g**, **2h** and **2**k show strongest fluorescence ability because of electron donating groups: -CH_3, -CF_3, -OEt, *n*-Bu, meanwhile, compounds **2e** owns an S atom and **2k** contains complex core of Fe elements.

When diluting solutions of 5 compounds **2d**, **2e**, **2g**, **2h** and **2k** into solutions with concentrations of 10^{-4}M and 10^{-5}M, the fluorescence intensity decreased compared to that at 10^{-3}M. The fluorescence spectra of the **2d**, **2e**, **2g**, **2h** and **2k** solutions at 10^{-4}M and 10^{-5} M are presented in Fig. 3 (a and b).

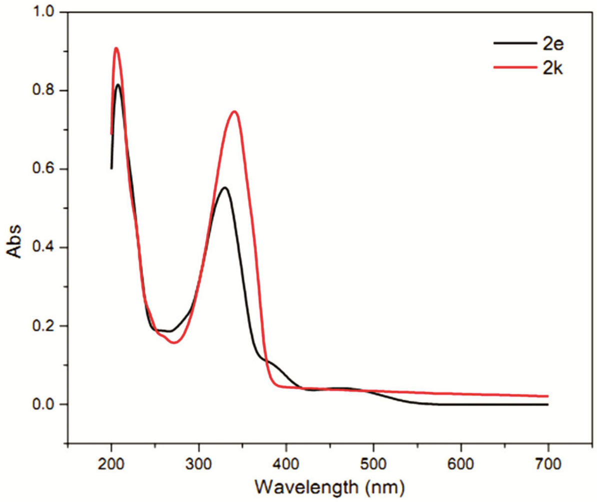


Fig. 1 — UV – Vis absorption of **2e** and **2k**

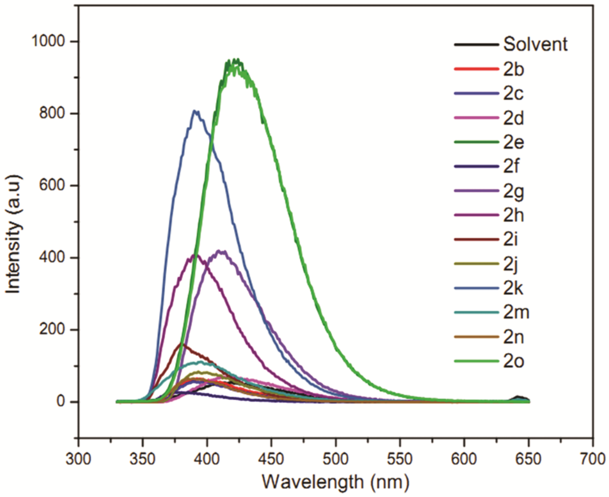


Fig. 2 — Fluorescene spectra of **2b** – **2o** solutions at 10^{-3}M

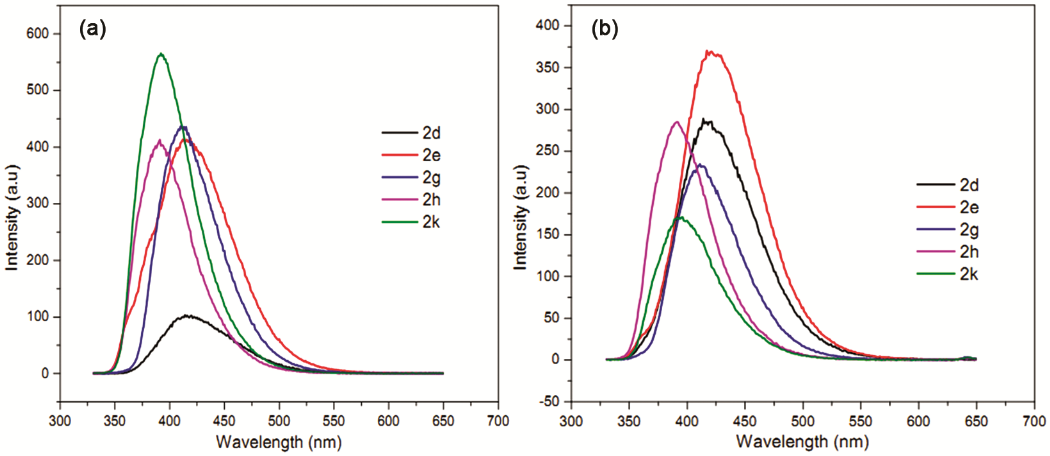


Fig. 3 — (a) The fluorescence spectra of **2d**, **2e**, **2g**, **2h** and **2k** solutions at 10^{-4}M; (b) The fluorescence spectra of **2d**, **2e**, **2g**, **2h** and **2k** solutions at 10^{-5}M

Conclusion

Assembling of 2-(4-bromophenyl)benzothiazole **1** and phenylboronic acid derivatives derived to form 15 compounds **2a-o** containing benzothiazole in up to 97% yield for from 17 to 48 hours. Compounds **2d**, **2e**, **2g**, **2h** and **2k** have the strongest fluorescence, especially **2e** and **2k** at 10^{-5}M and 10^{-4}M respectively.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

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