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The synthesis and properties of triazine-stilbene fluorescent brighteners containing a monophenolic antioxidant

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Abstract

Various triazine-stilbene fluorescent brighteners that were substituted with four different amine derivatives including a phenolic antioxidant on 4,4′-bistriazine moieties were synthesized. The obtained compounds (**6a–m**) were characterized by the analysis of proton NMR spectrum and confirmed by UV spectrum. The physical properties of the new compounds (**6a–m**) were determined by fastness test and whiteness measurement and compared with those of **CI86**.

Keywords: Antioxidant; Triazine-stilbene FBs; Non-symmetrical; Whiteness; Fastness; 4-(2-Aminoethyl)-2,6-di-*tert*-butylphenol

1. Introduction

Fluorescent brightener, which is currently being used in the fiber industry, holds higher stability for chemical bleaching since it is a high quality of whiteness and fastness [1]. It is possible to improve the fastness of fluorescent brightener by introducing various substituents. However, the whiteness is diminished and swept away when it is exposed to sunlight. It is assumed that the chemical skeleton of the fluorescent brightener is destroyed by free radical that is generated by sunlight. The solution for this problem is to develop new antioxidant fluorescent brightener, which inhibits the diminishment of whiteness due to prevention of autooxidation. Nowadays, our research interests are focused on the development of the fluorescent brightener that is capable of being exposed to the sunlight for a long time [2,3].

Since their usage as dyes for the first time in 1886, disodium 4,4′-diaminostilbene-2,2′-disulfonate derivatives are most widely used as the fluorescent brighteners for whitening both cotton and wool [4–8]. Previously, we reported disodium 4,4′-bis-(triazinylamino)stilbene-2,2′-disulfonate derivatives containing bisphenolic antioxidants (Fig. 1) which are symmetrically substituted on bistriazine moieties [2,3].

From the various physical tests, the compounds containing bisphenolic antioxidants showed lower degree of whiteness than did **CI86** and **CI90**, which are used for commercial purposes [3]. On the other hand, they preserved excellent light fastness that was assessed by using Xenon arc lamp method in accordance with ISO 105-B02. It is assumed that the lower whiteness resulted from the hydrophobic property of the phenolic antioxidant. For the development of the whiteness, therefore, it is necessary to increase the solubility of antioxidant fluorescent brighteners.

In this study, disodium 4-[2-RNH-4-{2-(2,6-di-*tert*-butylphenol)-ethylamino}-1,3,5-triazine-6-yl]amino-4′-(2-amino-4-anilino-1,3,5-triazine-6-yl)aminostilbene-2,2′-disulfonate derivatives, which are non-symmetrically substituted with various amines and 4-(2-aminoethyl)-2,6-di-*tert*-butylphenol (**5**) as a phenolic antioxidant on bistriazine moiety were synthesized as the non-symmetrically antioxidant fluorescent brighteners (**6a–m**). The structures of obtained compounds were characterized by the analysis of proton NMR spectrum and confirmed by UV

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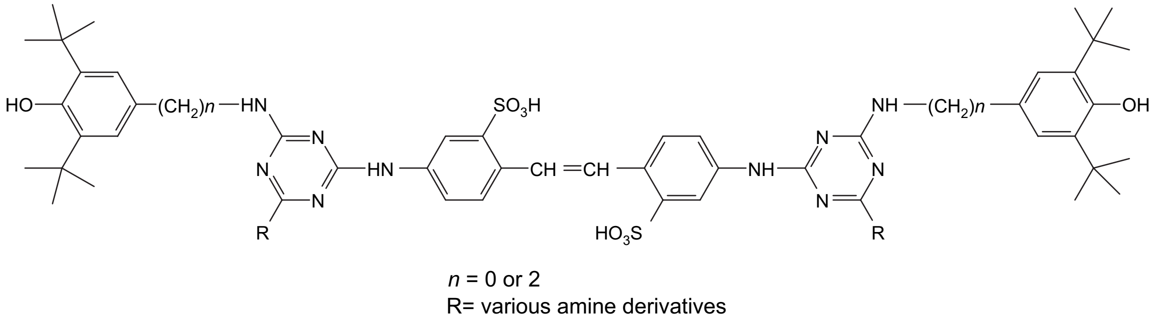


Fig. 1. The structure of 4,4′-bis(triazinylamino)stilbene-2,2′-disulfonic acid derivatives containing bisphenolic antioxidants.

spectrum. The physical properties were determined by various fastness test and whiteness measurement.

2. Result and discussion

2.1. Synthesis of dyes

Non-symmetrically substituted triazine-stilbene fluorescent brighteners containing the phenolic antioxidant (**6a–m**) were synthesized by the sequence shown in Scheme 1 and relevant data are given in Table 1.

The synthesis of the compounds **6a–m** was started from the reaction of 4-amino-4′-(2-amino-4-anilino-1,3,5-triazine-6-yl)aminostilbene-2,2′-disulfonic acid (**1**) [9] with 1 equiv of 2,4,6-trichloro-1,3,5-triazine (**2**). It is well known that three chloro groups of compound (**2**) show the different chemical reactivities with the nucleophiles depending on the reaction temperature [9,10].

After stirring 4-amino-4′-(2-amino-4-anilino-1,3,5-triazine-6-yl)aminostilbene-2,2′-disulfonic acid (**1**) with 1 equiv of 2,4,6-trichloro-1,3,5-triazine (**2**) at 0–5 °C, the resulting disodium 4-(2,4-dichloro-1,3,5-triazine-6-yl)amino-4′-(2-amino-4-anilino-1,3,5-triazine-6-yl)aminostilbene-2,2′-disulfonate (**3**) was treated with amine derivative at 30 °C without separation. The substitution of an aliphatic or an aromatic amine derivative with a second chloride on triazine moiety was established within 3–4 h.

Finally, 4-(2-aminoethyl)-2,6-di-*tert*-butyl-phenol (**5**) [3] was added *in situ* to the reaction mixture. The third substitution reaction occurred when the temperature reached 80 °C.

2.2. Assessment of colour and various fastness

Characteristics of the compounds (**6a–m**) are compared with those of **CI86**, which is used for commercial purposes. The chemical structure of **CI86** is shown in Fig. 2.

The newly synthesized compounds (**6a–m**) were applied at concentrations of 0.05%, 0.1%, 0.3%, 0.5%, and 1% relative to the weight of cotton fiber. The whiteness were determined according to the CIELAB system. The obtained data are presented with CIE L*, a*, b* coordinate and compared with those of **CI86** in Table 2. It is apparent that the compounds (**6a–m**) showed a lower degree of whiteness than **CI86**. The data of chlorinated water and light fastness are given in Table 3. The results show that the value of chlorinated water fastness in the compounds (**6a–m**) is the same as or higher than the value of **CI86**. Above all, the compound **6b** showed excellent fastness. The results show that the value of light fastness in the compounds (**6a–m**) is the same as or lower than the value of **CI86**. The compounds (**6a–m**) provided better rubbing fastness than **CI86** as shown in Table 3. Washing fastness data are given in Table 4. The results from the washing fastness test showed that all the compounds (**6a–m**) preserved excellent fastness, especially superior for cotton, than **CI86**.

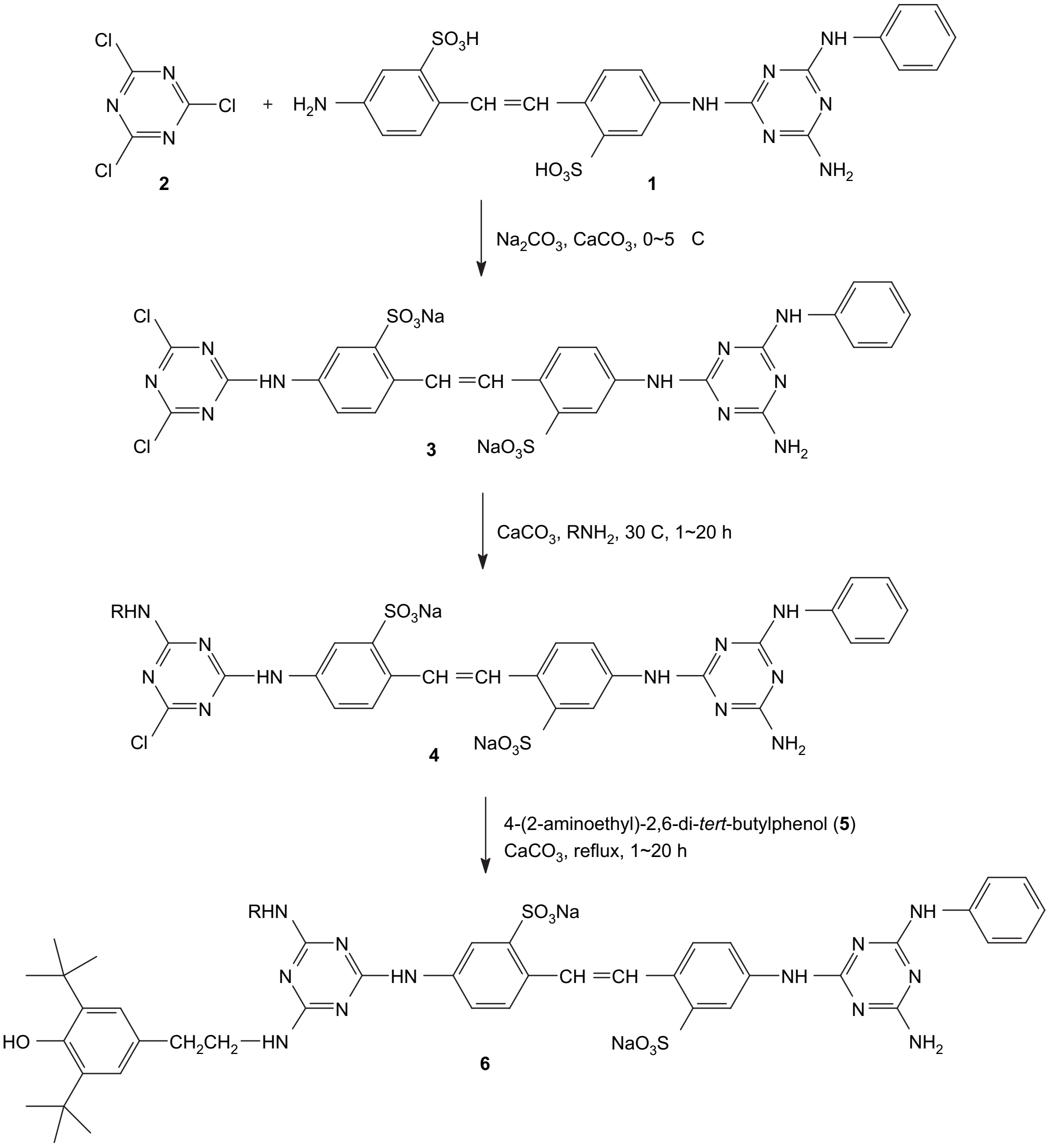
3. Conclusion

Various triazine-stilbene derivatives (**6a–m**) containing the monophenolic antioxidant were prepared as antioxidant fluorescent brighteners in good yield. On the basis of the results obtained from the various physical tests, compounds (**6a–m**) present the higher degrees of whiteness in comparison with those of the 4,4′-bis(triazinylamino)stilbene-2,2′-disulfonic acid derivatives containing bisphenolic antioxidants. On the other hand, compounds (**6a–m**) showed higher dyeing properties through various fastness tests than **CI86**, but provided the slightly lower degree of light fastness than **CI86**.

4. Experimental

4.1. Materials and analysis

All chemicals, which were purchased from commercial sources (Aldrich, Merck, Duksan, Yakuri, Junsei, etc.), were of analytical grade. The solvents were purified by distillation and the other reagents were used without further purification. The ^{1}H NMR spectrum was measured at 300 MHz using a Varian Mercury 300. The UV spectrum was obtained on a Shimadzu UV-2401PC. TLC was carried out using Merck silica gel plates (F254) with distilled solvents. Light fastness results were obtained on a Xenotest 150S. Washing fastness and chlorinated water fastness were tested on an Atras LP2. Rubbing fastness results were obtained on an Atras CM-5. The computer color matching (CCM) data were obtained on a SF600 Data color.



Scheme 1.

4.2. *General procedure for the preparation of triazine-stilbene fluorescent brighteners containing the phenolic antioxidant derivatives (**6a–m**)*

To a stirred suspension of 2,4,6-trichloro-1,3,5-triazine (**2**, 1.48 g, 8.0 mmol), $CaCO_3$ (0.40 g, 4.0 mmol) and small amounts of dispersant agent in water (140 mL) was added dropwise a solution of 4-amino-4′-(2-amino-4-anilino-1,3,5-triazine-6-yl)amino-stilbene-2,2′-disulfonic acid (**1**, 4.44 g, 8.0 mmol) and Na_2CO_3 (0.85 g, 8.0 mmol) in water (140 mL) in the temperature range of 0–5 °C. After the addition, the reaction mixture was stirred for 2 h below 5 °C. To the reaction mixture were added $CaCO_3$ (0.40 g, 4.0 mmol), antifoaming agent and amine derivative (8.0 mmol), and then allowed to warm to 30 °C. After being stirred at 30 °C for 1–20 h, $CaCO_3$ (0.40 g, 4.0 mmol) and phenolic derivative (**1**, 8.0 mmol) were added portionwise to the resulting reaction mixture. After being heated under reflux for 3 h, the reaction mixture was allowed to cool to room temperature. The precipitate was filtered and dried.

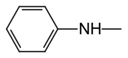
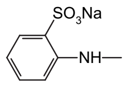
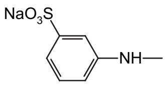
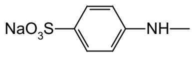
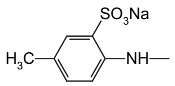
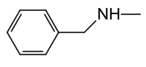
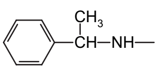
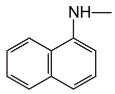
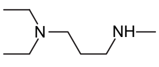
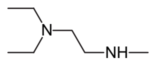
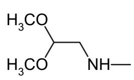
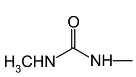
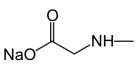
4.2.1. *Synthesis of compound **6a***

Yields 84%, ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.79 (t, 2H, $-CH_2$), 3.50 (s, 2H, $-CH_2$), 6.59 (s, 1H, –OH), 6.70 (s, 2H, ArH), 6.92 (d, 4H, ArH), 7.23 (t, 6H, ArH), 7.57 (d, 2H, ArH), 7.77 (s, 2H, –CH=CH–), 7.82 (d, 2H, ArH), 7.98 (s, 2H, ArH), 8.29 (s, 1H, –NH), 9.03 (s, 2H, –NH), 9.13 (s, 2H, –NH).

4.2.2. *Synthesis of compound **6b***

Yields 47%; ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.76 (t, 2H, $-CH_2$), 3.57 (s, 2H, $-CH_2$), 6.61 (s, 1H, –OH), 6.8 (m, 4H, ArH), 6.95 (s, 2H, ArH), 7.24 (t, 5H, ArH), 7.57 (d, 2H, ArH), 7.80 (d, 2H, ArH), 7.83 (s, 2H, –CH=CH–), 7.97 (s, 2H, ArH), 8.20 (s, 1H, –NH), 9.04 (s, 1H, –NH), 9.16 (s, 1H, –NH), 9.64 (s, 1H, –NH).

Table 1
The obtained yields and UV spectum data of compound (**6a–m**)

Entry	R–NH–	Yield (%)	UV (λ_{max} = nm/log ε)
6a		84	354(4.59)
6b		47	353(4.78)
6c		89	351(4.36)
6d		98	353(4.44)
6e		93	351(4.70)
6f		85	352(4.81)
6g		94	352(3.88)
6h		88	352(4.78)
6i		91	352(4.59)
6j		80	352(3.95)
6k		98	361(4.47)
6l		70	351(3.96)
6m		67	353(4.68)

4.2.3. Synthesis of compound **6c**

Yields 89%; ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.77 (t, 2H, $-CH_2$), 3.50 (s, 2H, $-CH_2$), 6.60 (s, 1H, $-OH$), 6.88 (m, 2H, ArH), 6.94 (s, 2H, ArH), 7.16 (m, 3H, ArH), 7.23 (t, 3H, ArH), 7.57 (d, 2H, ArH), 7.77 (s, 2H, $-CH{=}CH-$), 7.82 (d, 2H, ArH), 7.98 (s, 3H, ArH), 8.18 (s, 2H, $-NH$), 9.03 (s, 2H, $-NH$), 9.14 (s, 1H, $-NH$).

4.2.4. Synthesis of compound **6d**

Yields 98%; ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.78 (t, 2H, $-CH_2$), 3.50 (s, 2H, $-CH_2$), 6.60 (s, 1H, $-OH$), 6.86 (d, 2H, ArH), 6.96 (s, 2H, ArH), 7.24 (t, 3H, ArH), 7.58 (d, 2H, ArH), 7.76 (s, 2H, $-CH{=}CH-$), 7.83 (d, 2H, ArH), 7.98 (s, 2H, ArH), 8.23 (s, 2H, $-NH$), 9.03 (s, 2H, $-NH$), 9.14 (s, 1H, $-NH$).

4.2.5. Synthesis of compound **6e**

Yields 93%; ^{1}H NMR (DMSO-d_6) δ 1.16 (s, 3H, $-CH_3$), 1.36 (s, 18H, *tert*-butyl), 2.69 (m, 2H, $-CH_2$), 3.56 (s, 2H, $-CH_2$), 6.63 (s, 1H, $-OH$), 6.90 (d, 2H, ArH), 6.96 (s, 2H, ArH), 7.25 (t, 3H, ArH), 7.58 (d, 3H, ArH), 7.77 (s, 2H, $-CH{=}CH-$), 7.84 (d, 3H, ArH), 7.97 (s, 3H, ArH), 8.24 (s, 1H, $-NH$), 9.07 (s, 2H, $-NH$), 9.18 (s, 2H, $-NH$).

4.2.6. Synthesis of compound **6f**

Yields 85%; ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.70 (t, 2H, $-CH_2$), 3.47 (s, 2H, $-CH_2$), 6.61 (s, 1H, $-OH$), 6.94 (s, 2H, ArH), 7.24 (t, 6H, ArH), 7.34 (d, 4H, ArH), 7.58 (d, 2H, ArH), 7.77 (s, 2H, $-CH{=}CH-$), 7.84 (d, 2H, ArH), 7.99 (s, 3H, ArH), 8.24 (s, 1H, $-NH$), 9.05 (s, 2H, $-NH$), 9.20 (s, 2H, $-NH$).

4.2.7. Synthesis of compound **6g**

Yields 94%; ^{1}H NMR (DMSO-d_6) δ 1.36 (s, 18H, *tert*-butyl), 2.58 (d, 3H, $-CH_3$), 2.75 (t, 2H, $-CH_2$), 3.05 (m, 2H, $-CH_2$), 5.21 (s, 1H, $-CH$), 6.60 (s, 1H, $-OH$), 6.93 (s, 2H, ArH), 6.90 (d, 4H, ArH), 7.21 (m, 6H, ArH), 7.57 (d, 2H, ArH), 7.74 (s, 2H, $-CH{=}CH-$), 7.83 (d, 2H, ArH), 7.97 (s, 2H, ArH), 8.24 (s, 1H, $-NH$), 9.02 (s, 2H, $-NH$), 9.13 (s, 2H, $-NH$).

4.2.8. Synthesis of compound **6h**

Yields 88%; ^{1}H NMR (DMSO-d_6) δ 1.35 (s, 18H, *tert*-butyl), 2.67 (t, 2H, $-CH_2$), 3.50 (m, 2H, $-CH_2$), 6.59 (s, 1H, $-OH$), 6.78 (s, 2H, ArH), 6.88 (s, 2H, ArH), 7.23 (t, 3H, ArH), 7.49 (m, 5H, ArH), 7.60 (d, 2H, ArH), 7.70 (d, 2H, ArH), 7.74 (s, 2H, $-CH{=}CH-$), 7.83 (d, 2H, ArH), 7.96 (s, 2H, ArH), 8.23 (s, 1H, $-NH$), 9.02 (s, 2H, $-NH$), 9.13 (s, 2H, $-NH$).

4.2.9. Synthesis of compound **6i**

Yields 91%; ^{1}H NMR (DMSO-d_6) δ 1.14 (s, 6H, $-CH_3$), 1.37 (s, 18H, *tert*-butyl), 2.71 (s, 4H, $-CH_2$), 3.05 (m, 2H, $-CH_2$), 3.50 (m, 2H, $-CH_2$), 3.51 (s, 6H, $-CH_2$), 6.70 (s, 1H, $-OH$), 6.85 (d, 2H, ArH), 6.92 (s, 2H, ArH), 7.23 (t, 3H, ArH), 7.53 (d, 2H, ArH), 7.74 (s, 2H, $-CH{=}CH-$),

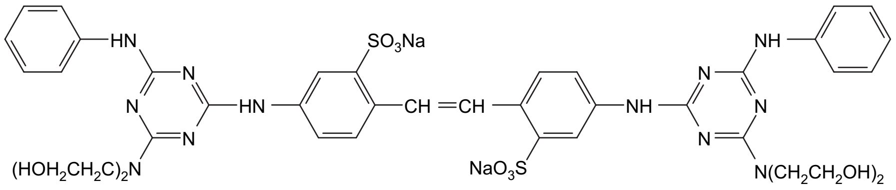


Fig. 2. The structure of **CI86**.

7.82 (d, 2H, ArH), 7.96 (s, 2H, ArH), 8.23 (s, 2H, −NH), 9.02 (s, 2H, −NH), 9.14 (s, 1H, −NH).

*4.2.10. Synthesis of compound **6j***

Yields 80%; ^{1}H NMR (DMSO-d_6) δ 1.01 (d, 6H, −CH$_3$), 1.36 (s, 18H, *tert*-butyl), 2.57 (m, 4H, −CH$_2$), 2.71 (m, 2H, −CH$_2$), 3.40 (s, 2H, −CH$_2$), 3.50 (s, 4H, −CH$_2$), 6.60 (s, 1H, −OH), 6.91 (d, 2H, ArH), 6.92 (s, 2H, ArH), 7.23 (t, 3H, ArH), 7.56 (d, 2H, ArH), 7.75 (s, 2H, −CH=CH−), 7.82 (d, 2H, ArH), 7.96 (s, 2H, ArH), 8.22 (s, 2H, −NH), 9.02 (s, 2H, −NH), 9.14 (s, 1H, −NH).

*4.2.11. Synthesis of compound **6k***

Yields 98%; ^{1}H NMR (DMSO-d_6) δ 1.23 (s, 6H, −CH$_3$), 1.36 (s, 18H, *tert*-butyl), 2.72 (s, 2H, −CH$_2$), 3.50 (s, 2H, −CH$_2$), 3.50 (s, 2H, −CH$_2$), 4.55 (d, 2H, −CH$_2$), 6.60 (s, 1H, −OH), 6.90 (d, 2H, ArH), 6.94 (s, 2H, ArH), 7.24 (t, 3H, ArH), 7.58 (d, 2H, ArH), 7.76 (s, 2H, −CH=CH−), 7.84 (d, 2H, ArH), 7.98 (s, 2H, ArH), 8.24 (s, 1H, −NH), 9.04 (s, 2H, −NH), 9.15 (s, 2H, −NH).

*4.2.12. Synthesis of compound **6l***

Yields 70%; ^{1}H NMR (DMSO-d_6) δ 23 (d, 2H, −CH$_3$), 1.36 (s, 18H, *tert*-butyl), 2.69 (t, 2H, −CH$_2$), 3.44 (m,

Table 2
Colour fastness of cotton fiber dyed with compounds (**6a**−**m**)

Entry	o.w.f (%)	Whiteness	*L**	*a**	*b**	Entry	o.w.f (%)	Whiteness	*L**	*a**	*b**
CI86	0.05	123.69	91.91	2.55	−9.06	**6g**	0.05	88.68	92.05	0.49	−1.66
	0.1	132.07	91.77	2.78	−10.75		0.1	87.79	92.25	0.27	−1.37
	0.3	141.05	95.52	2.17	−12.00		0.3	76.75	91.68	−0.08	0.72
	0.5	142.28	92.85	1.52	−11.90		0.5	77.66	92.03	−0.24	0.69
	1	138.00	93.02	0.22	−10.56		1	73.65	91.88	−0.77	1.47
6a	0.05	98.47	92.21	1.06	−3.67	**6h**	0.05	90.76	91.22	1.17	−2.48
	0.1	101.58	92.18	1.18	−4.35		0.1	84.82	91.05	0.68	−1.29
	0.3	100.30	92.17	1.08	−4.08		0.3	84.14	91.32	0.32	−1.01
	0.5	94.45	92.26	0.51	−2.78		0.5	82.38	90.92	0.43	−0.82
	1	88.56	91.87	0.10	−1.70		1	64.83	90.00	0.66	2.44
6b	0.05	92.54	91.66	1.43	−2.66	**6i**	0.05	95.74	92.53	0.86	−2.95
	0.1	94.72	92.21	0.73	−2.87		0.1	91.99	92.21	0.61	−2.29
	0.3	97.06	92.27	0.71	−3.34		0.3	83.78	92.10	−0.07	−0.58
	0.5	99.00	92.08	1.14	−3.85		0.5	77.33	91.95	−0.44	0.72
	1	97.19	92.46	0.12	−3.29		1	59.87	90.67	−0.81	3.77
6c	0.05	96.00	92.42	0.91	−3.05	**6j**	0.05	97.69	92.27	0.95	−3.48
	0.1	99.90	91.98	1.87	−4.09		0.1	100.99	92.58	1.06	−4.05
	0.3	99.44	92.09	1.54	−3.93		0.3	89.08	92.18	0.17	−1.67
	0.5	99.19	92.38	0.83	−3.75		0.5	86.95	91.19	−0.02	−1.34
	1	92.63	92.10	0.43	−2.47		1	76.70	91.01	0.92	0.43
6d	0.05	94.21	92.02	1.00	−2.85	**6k**	0.05	94.66	91.66	1.50	−3.11
	0.1	97.14	91.97	1.36	−3.50		0.1	95.39	92.08	1.28	−3.06
	0.3	95.17	91.96	1.01	−3.07		0.3	89.01	91.54	1.48	−1.94
	0.5	91.69	91.49	1.50	−2.54		0.5	79.02	90.76	1.98	−0.16
	1	95.09	92.47	0.13	−2.82		1	65.90	90.31	2.56	2.42
6e	0.05	89.92	92.37	0.73	−1.78	**6l**	0.05	79.43	92.23	−0.88	0.38
	0.1	94.31	92.41	0.97	−2.69		0.1	79.33	91.99	0.03	0.29
	0.3	94.83	92.35	1.01	−2.82		0.3	72.12	91.98	−0.68	1.83
	0.5	91.05	92.51	0.86	−1.93		0.5	62.26	90.96	−0.04	3.42
	1	79.39	91.86	0.92	0.26		1	50.53	91.51	−1.88	6.16
6f	0.05	95.74	91.90	1.30	−3.23	**6m**	0.05	89.98	91.62	0.81	−2.13
	0.1	97.81	92.12	1.30	−3.57		0.1	91.73	91.75	0.89	−2.44
	0.3	99.76	91.97	1.47	−4.05		0.3	96.50	92.04	1.03	−3.33
	0.5	100.43	91.59	1.81	−4.36		0.5	95.72	91.89	1.07	−3.22
	1	92.42	91.98	0.73	−2.47		1	93.03	92.02	0.86	−2.59

Table 3
Various fastness of cotton fiber dyed with compounds (**6a–m**)

Compound	Chlorinated water fastness	Light fastness	Rubbing fastness	
			Dry	Wet
CI86	3	2	3–4	2
6a	3	1	4–5	3
6b	4	1	4–5	3–4
6c	3	1	4–5	3–4
6d	2	1	4–5	3–4
6e	1–2	2	4–5	2–3
6f	2–3	2	4–5	2–3
6g	1–2	1	4–5	4–5
6h	1–2	1	4–5	4
6i	1–2	1	4–5	4–5
6j	2	1	4–5	4–5
6k	1–2	2	4–5	3–4
6l	2	2	4–5	4–5
6m	2–3	2	4–5	3

Table 4
Washing fastness of cotton fiber dyed with compounds (**6a–m**)

Compound	Acetate	Cotton	Nylon	PET	Acrylic	Wool
CI86	4–5	1	3	4–5	4–5	4–5
6a	4–5	3	4–5	4–5	4–5	4–5
6b	4–5	1–2	4–5	4–5	4–5	4–5
6c	4–5	2	4–5	4–5	4–5	4–5
6d	4–5	3	4–5	4–5	4–5	4–5
6e	4–5	2	4–5	4–5	4–5	4–5
6f	4–5	1–2	4–5	4–5	4–5	4–5
6g	4–5	4–5	4–5	4–5	4–5	4–5
6h	4–5	4	4–5	4–5	4–5	4–5
6i	4–5	3	4–5	4–5	4–5	4–5
6j	4–5	1–2	4–5	4–5	4–5	4–5
6k	4–5	3	4–5	4–5	4–5	4–5
6l	4–5	4–5	4–5	4–5	4–5	4–5
6m	4–5	2	4–5	4–5	4–5	4–5

2H, $-CH_2$), 6.61 (s, 1H, $-OH$), 6.89 (s, 2H, ArH), 6.92 (d, 2H, ArH), 7.23 (t, 3H, ArH), 7.56 (d, 2H, ArH), 7.75 (s, 2H, $-CH{=}CH-$), 7.83 (d, 2H, ArH), 7.96 (s, 2H, ArH), 8.25 (s, 2H, $-NH$), 8.76 (s, 1H, $-NH$), 9.02 (s, 2H, $-NH$), 9.13 (s, 1H, $-NH$).

4.2.13. Synthesis of compound *6m*

Yields 67%; ^{1}H NMR (DMSO-d_6) δ 1.18 (s, 2H, $-CH_2$), 1.36 (s, 18H, *tert*-butyl), 2.72 (s, 2H, $-CH_2$), 3.64 (m, 2H, $-CH_2$), 6.61 (s, 1H, $-OH$), 6.91 (d, 2H, ArH), 6.95 (s, 2H, ArH), 7.25 (t, 3H, ArH), 7.58 (d, 2H, ArH), 7.76 (s, 2H, $-CH{=}CH-$), 7.84 (d, 2H, ArH), 7.98 (s, 2H, ArH), 8.25 (s, 1H, $-NH$), 9.05 (s, 2H, $-NH$), 9.16 (s, 2H, $-NH$).

4.3. Dyeing properties

4.3.1. Dyeing procedure

Dyeing of cotton was carried out precisely at 80 °C for 1 h. This was done with a paste of finely powdered dye, 0.1% aqueous NaOH solution and water at 40 °C, and the temperature was raised to 80 °C for cotton at the rate of 1 °C/min.

4.3.2. Colour fastness test

The light fastness test was assessed by using Xenon arc lamp method in accordance with ISO 105-B02. The rubbing fastness test was carried out using crock meter method in accordance with ISO 105-X12 and the wash fastness test in accordance with ISO 105-C01 and chlorinated water fastness test in accordance with KS K 0725. The results are given in Tables 3 and 4.

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References

[1] Williamson R. Fluorescent brightening agents. Amsterdam: Elsevier; 1980.

[2] Lee JK, Um SI, Kang YH, Baek DJ. Dyes and Pigments 2005;64:25–30.

[3] Um SI, Lee JK, Kang YH, Baek DJ. Dyes and Pigments 2005;64:93–9.

[4] Findley WR. Journal of the American Oil Chemists' Society 1968;65:679–83.

[5] Milligan B, Holt A. Australian Journal of Chemistry 1974;27:195–203.

[6] Leaver IH, Milligan B. Dyes and Pigments 1984;5:109–44.

[7] Alsins J, Bjorling M, Furo I, Egle V. Journal of Physical Organic Chemistry 1999;12:171–5.

[8] Modi BR, Naik NM, Naik DN, Desai KR. Dyes and Pigments 1993;23:65–72.

[9] Hoog P, Gomez P, Driessen W, Reedijk J. Tetrahedron Letters 2002;43:6783–6.

[10] Yang X, Lowe CR. Tetrahedron Letters 2003;44:1359–62.