

Studies on Synthesis and Dyeing Performance of Some Fluorescent Brightening Agents Based on Stilbene Derivatives

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Various fluorescent brightening agents have been synthesised by condensing 4,4'-diaminostilbene-2,2'-disulphonic acid with cyanuric chloride, N-benzyl aniline and various amines. They are characterised by their nitrogen elemental analysis, UV-visible spectra, IR and PMR spectra. Their dyeing performance has been assessed on polyester and nylon fabric to show very good to excellent wash fastness and good to very good light fastness properties.

INTRODUCTION

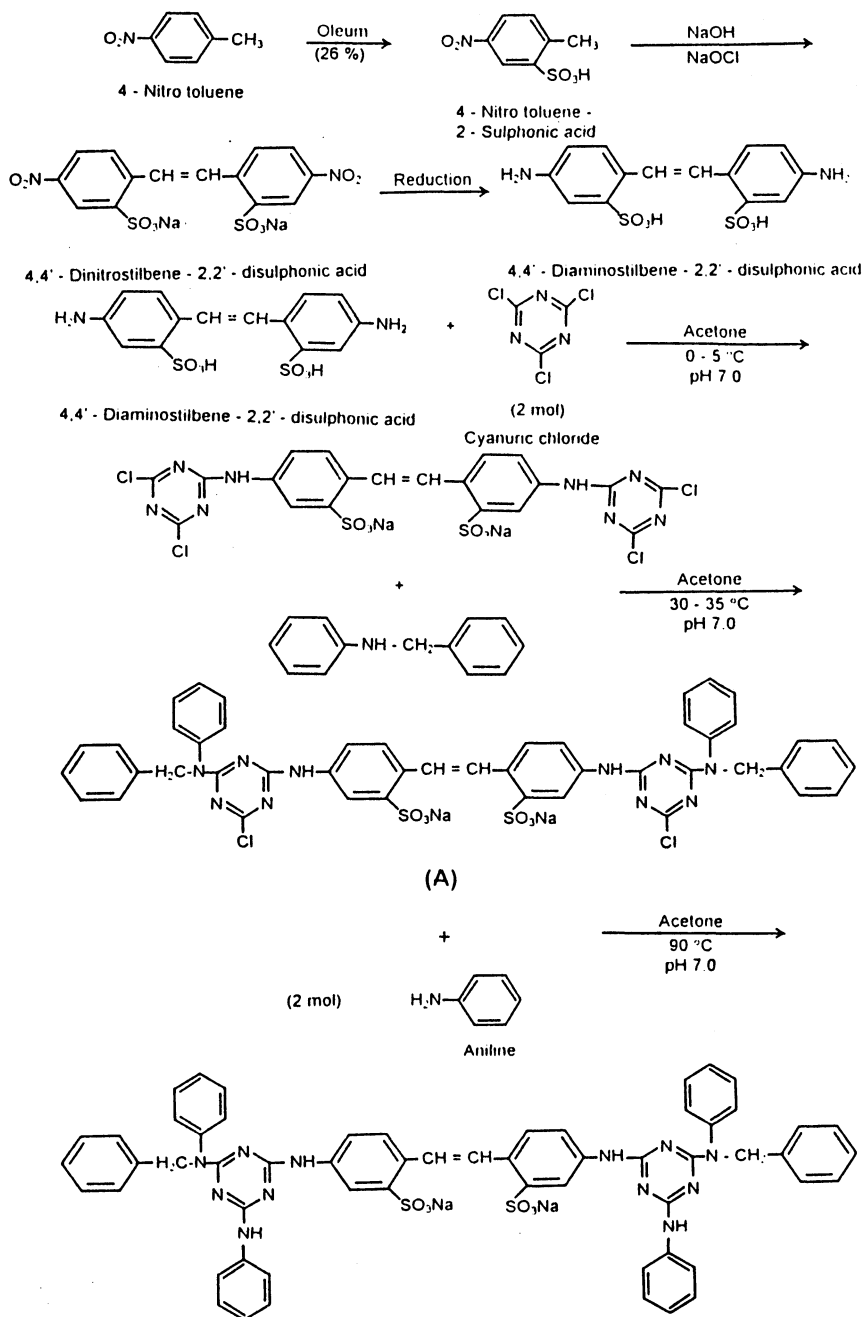
Fluorescent Brightening Agents (FBA) are colourless fluorescent dyes¹ and their mode of application to the various substrates parallels that of the appropriate dyes². They are applied to textiles to reduce yellowness and to increase fabric brightness³. They function by absorbing ultra-violet radiations present in the light source and re-emitting it as visible blue light. When first introduced into the textile industry, fluorescent brightening agents were used on cellulosics⁴. But today, they are frequently being applied on various textile fibres, including cellulosics, during manufacturing and are also incorporated into laundry detergents⁵. The fluorescent brightening agents are mainly derived from stilbene⁶ and coumarin derivatives⁷.

EXPERIMENTAL

Synthesis of 4,4'-bis-[4-chloro-6-(N-benzylanilino)-s-triazin-2-yl-amino]-stilbene-2,2'-disulphonic acid (A)

Cyanuric chloride (3.7 g, 0.02 mole) in acetone (40 mL) was stirred around 5°C for a period of 1 h. A neutral solution of 4,4'-diaminostilbene-2,2'-disulphonic acid⁸ (3.7 g, 0.01 mole) in sodium carbonate solution (10% w/v), which was previously cooled below 5°C, was added in small lots at about 5°C. The reaction mass was then stirred for 3 h to a negative amine test.

To the above clear solution, N-benzylaniline⁹ (3.66 g, 0.02 mole) dissolved in sodium carbonate (10% w/v) was added. The reaction mixture was stirred mechanically for 5 h at 35°C, maintaining pH 7 with sodium carbonate solution (10% w/v). The clear solution obtained was used for subsequent reaction.



Formation of FBA-1 to FBA-16

Various amines (R) (0.02 mol) (Table-1) dissolved in acetone (20 mL) were added to a clear solution of 4,4'-bis[4-chloro-6-(N-benzylanilino)-s-triazin-2-yl-amino]-stilbene-2,2'-disulphonic acid (A). The mixture was refluxed at 90°C for 3 h, maintaining pH 7 with sodium carbonate solution (10% w/v). The reaction mixture was cooled and the product was filtered and dried. The resultant fluorescent brightening agent was purified by dissolving in DMF and reprecipitated by adding acetone.

Dyeing of fibre: All the fluorescent brightening agents were applied on polyester and nylon by using standard procedure¹⁰.

RESULTS AND DISCUSSION

The UV-visible spectra of all fluorescent brightening agents were recorded on Beckmann DB-GT Grating Spectrophotometer. The IR spectra were recorded on a Perkin-Elmer Model-377 spectrophotometer using KBr pellets. Fastness to light was assessed in accordance with BS: 1006-1978 and the wash fastness test in accordance with IS: 765-1979. The characterisation data of fluorescent brightening agents are given in Table-1.

TABLE-1
CHARACTERISATION OF FLUORESCENT BRIGHTENING AGENTS

FBA No.	R—H	m.f.	m.w.	Yield (%)	m.p. (°C) (d)	N (%)	
						Found	Required
FBA-1	Aniline	C ₅₈ H ₄₆ O ₆ N ₁₂ S ₂ Na ₂	1146	72.0	285	15.00	15.05
FBA-2	<i>o</i> -Toluidine	C ₆₀ H ₅₀ O ₆ N ₁₂ S ₂ Na ₂	1144	81.0	310	14.66	14.68
FBA-3	<i>m</i> -Toluidine	C ₆₀ H ₅₀ O ₆ N ₁₂ S ₂ Na ₂	1144	80.0	270	14.66	14.68
FBA-4	<i>p</i> -Toluidine	C ₆₀ H ₅₀ O ₆ N ₁₂ S ₂ Na ₂	1144	78.0	295	14.64	14.68
FBA-5	<i>o</i> -Nitroaniline	C ₅₈ H ₄₄ O ₁₀ N ₁₄ S ₂ Na ₂	1206	77.0	284	16.21	16.25
FBA-6	<i>m</i> -Nitroaniline	C ₅₈ H ₄₄ O ₁₀ N ₁₄ S ₂ Na ₂	1206	76.5	302	16.20	16.25
FBA-7	<i>p</i> -Nitroaniline	C ₅₈ H ₄₄ O ₁₀ N ₁₄ S ₂ Na ₂	1206	74.6	265	16.24	16.25
FBA-8	<i>o</i> -Chloroaniline	C ₅₈ H ₄₄ O ₆ N ₁₂ S ₂ Cl ₂ Na ₂	1185	81.0	292	14.16	14.17
FBA-9	<i>m</i> -Chloroaniline	C ₅₈ H ₄₄ O ₆ N ₁₂ S ₂ Cl ₂ Na ₂	1185	83.0	277	14.14	14.17
FBA-10	<i>p</i> -Chloroaniline	C ₅₈ H ₄₄ O ₆ N ₁₂ S ₂ Cl ₂ Na ₂	1185	84.5	289	14.16	14.17
FBA-11	<i>o</i> -Anisidine	C ₆₀ H ₅₀ O ₈ N ₁₂ S ₂ Na ₂	1176	79.0	297	14.26	14.28
FBA-12	<i>p</i> -Anisidine	C ₆₀ H ₅₀ O ₈ N ₁₂ S ₂ Na ₂	1176	78.5	312	14.26	14.28
FBA-13	Ammonia	C ₄₆ H ₃₈ O ₆ N ₁₂ S ₂ Na ₂	964	81.0	275	17.38	17.42
FBA-14	Benzylamine	C ₆₀ H ₅₀ O ₆ N ₁₂ S ₂ Na ₂	1144	79.0	288	14.56	14.58
FBA-15	Morpholine	C ₅₄ H ₅₀ O ₈ N ₁₂ S ₂ Na ₂	1104	76.0	276	15.20	15.21
FBA-16	Piperidine	C ₅₆ H ₅₄ O ₆ N ₁₂ S ₂ Na ₂	1100	74.6	291	15.24	15.27

All the FBAs, in general showed: C—N stretching vibration at 800–810 cm^{-1} for s-triazine, —CH=CH— stretching vibration at 1020 cm^{-1} for —CH=CH—, S=O stretching vibration at 1220 cm^{-1} for sulphonic acid, N—H stretching vibration at 3410–3400 cm^{-1} for secondary amine.

The PMR spectra (300 MHz, CDCl_3 + DMSO-d_6) of the FBA-1 showed signals at 2.896 (4H, — CH_2), 3.51 (6H, DMSO-d_6 solvent), 6.627–7.003 (2H, —CH=), 7.296–7.835 (3 and 6 H, aromatic protons) and 10.7 (4H, —NH).

Fastness properties: The data of fastness properties show that the light fastness ranges from good to very good for both the fibres. The fastness to washing ranges from very good to excellent for both the fibres. This indicates good substantivity of these FBAs for the fibres (Table-2).

TABLE-2
FASTNESS PROPERTIES OF FBAs

FBA No.	λ_{max}	Light fastness		Wash fastness	
		Polyester	Nylon	Polyester	Nylon
FBA-1	348	5	5–6	4	4
FBA-2	334	5	5	4–5	4–5
FBA-3	340	5–6	5	5	4
FBA-4	346	6	6	4	4
FBA-5	352	5	6	4	4
FBA-6	342	5	5–6	4–5	5
FBA-7	340	4–5	5	4	4–5
FBA-8	332	6	5	4–5	5
FBA-9	325	5	5	4	4
FBA-10	338	5–6	5–6	5	4
FBA-11	356	5	5–6	4	4
FBA-12	347	5	5	4	4–5
FBA-13	351	5–6	5	5	4
FBA-14	342	6	5–6	4–5	4
FBA-15	357	5	5	4	4–5
FBA-16	340	5	6	4	4

Light fastness : 1-poor, 2-slight, 3-moderate, 4-fair, 5-good, 6-very good, 7-excellent, and 8-maximum.

Wash fastness : 1-poor, 2-fair, 3-good, 4-very good and 5-excellent.

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