

Symmetrically Substituted 4,4'-Bis-(1,3,5-Triazinylamino)Stilbene-2,2'-Disulfonate Derivatives as Fluorescent Brighteners

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Abstract: Disodium 4,4'-bis-(1,3,5-triazinylamino)stilbene-2,2'-disulfonate derivatives were synthesized by reacting 4,4'-diaminostilbene-2,2'-disulfonic acid with 2,4,6-trichloro-1,3,5-triazine. The resulting dichloro-sym-triazinyl intermediate was further condensed in two steps with amines by nucleophilic substitutions reaction on their triazine moiety to furnish the fluorescent brighteners. The structures of these compounds were characterized by using spectroscopic techniques i.e., ¹H-NMR, FT-IR, UV and elemental analysis. These compounds were used as whitening agents of 100% cotton fiber and their performance was evaluated by light fastness, chlorinated water fastness, washing fastness and CIE whiteness measurements.

Keywords: Fluorescent brightener, whiteness, fastness, cotton.

1. INTRODUCTION

Fluorescent brighteners (FBs) have become a part of everyday life and culture; these agents have found an increasing use in diverse fields and they are widely used in the textile industry in order to improve the whiteness properties of a large range of textile fiber materials. They increased the whiteness by re-emitting more visible light than correspond to the absorbed light; a transformation of the part of light from the ultraviolet region into visible spectrum i.e. absorption around 360 nm, and fluorescence in the VIS, with a typical maximum at 430-440 nm wavelength [1,2]. One important class of FBs is 4,4'-bis-(triazinylamino)stilbene-2,2'-disulfonate derivatives which are widely used to create intense and bright white shades in textiles, pulps and papers, and as additives to laundry detergents. Many such compounds are known and a number of them are commercially available [3-12]. Nowadays, some of these compounds are also used as ultraviolet absorbing agents (UVAs) to the textile material. In some areas of the world, e.g. Australia and America where people enjoy especially sunny climates, there has been a great increase in the awareness of the potential hazards of undue exposure to sunlight. One aspect of the desire to increase the sun protection factor (SPF), they are used on the most natural and synthetic textile materials, especially lightweight summer fabrics, to provide skin beneath clothing protection against Ultraviolet A (UV-A or Long Wave or Black Light) and Ultraviolet B (UV-B or Medium Wave) ranges radiation [13-16]. In this paper, we report the synthesis of a series of triazine derivatives of 4,4'-bis-(1,3,5-triazinylamino)stilbene-2,2'-disulfonate and their

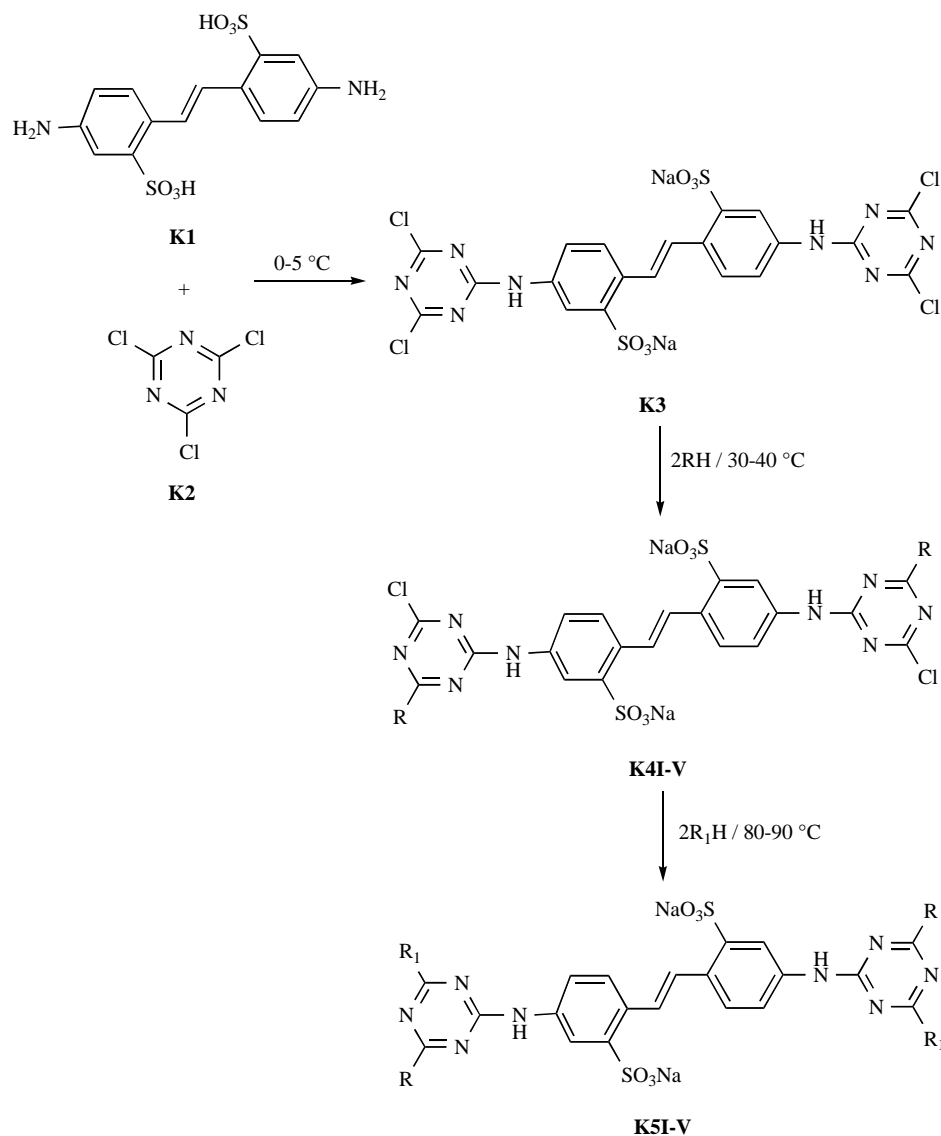
performance was evaluated by light fastness, chlorinated water fastness, washing fastness and CIE whiteness measurements.

2. RESULTS AND DISCUSSION

2.1. Synthesis

Triazine-stilbene brighteners were typically prepared by following three-step process, summarized in Fig. (1). Where by two moles of 2,4,6-trichloro-1,3,5-triazine were reacted with 1 mole of 4,4'-diaminostilbene-2,2'-disulfonic acid at 0-5 °C, while maintaining pH at 4.5-5.5 by the addition of 20% (w/v) aqueous sodium carbonate. The reaction mixture was stirred for 1 hour while testing for the disappearance of 4,4'-diaminostilbene-2,2'-disulfonic acid by TLC and iodine spot test. The reaction flask was then removed from ice bath and the temperature was raised up to 20 °C and 2 mol of respective amine (RH) was added gradually and stirred for 2-3 h while maintaining pH 7. The product from step-2 (mono-chloro-sym-triazinyl) was isolated by salting out and collected through vacuum filtration; the wet cake was used without drying in the next step. Into a 250 mL flask equipped with pH meter and thermometer was charged with wet cake, a step-2 reaction mixture followed by 150 mL of de-ionized water. To the stirred mixture was added 2 mol (with respect to starting material) of the respective amines (R₁H) followed by a rise in temperature to 90 °C. The mixture was stirred for 3-5 hours at pH 8 and then cooled to ambient temperature. The product was isolated by the addition of sodium chloride. The reaction course was monitored by TLC. During the preparation of stilbene-triazine compounds it is necessary to take careful control of pH to avoid formation of unwanted hydrolyzed by-products. These impurities reduce the whiteness and make it hard to remove. The introduction of different nucleophiles to triazine moiety the reactivity of each chlorine replaced was decreased at each replacement stage due to a decrease in electropositive character of triazine

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Compound	R	R ₁
K5I	aniline	benzohydrazide
K5II	aniline	p-tolyl-hydrazide
K5III	m-aminosulfonic acid	benzohydrazide
K5IV	p-toluidine	benzohydrazide
K5V	m-aminosulfonic acid	p-tolyl-hydrazide

Fig. (1). Synthetic pathway for the compounds (**K5I-V**) and their structures.

moiety. The three chloro groups of 2,4,6-trichloro-1,3,5-triazine moiety showed different reactivity depending on the nucleophilicities and temperature. All the FBs treatments improved the whiteness of the cotton fabric, resulting in an increase in whiteness. The untreated cotton had a whiteness index of 80.43, whereas those for the FBs treated specimens ranged from 114.35 to 137.55. Compounds K5I, K5III and K5V showed a high degree of whiteness with a weak violet hue. The effect of the whiteness increase as the amount of FBs on substrate increases until a maximum value is reached. However, further increase in concentration of the

FBs results in a reduction in whiteness and total fluorescence, in general, due to an increase in the aggregation of the FBAs on the substrate, resulting in a shift in fluorescence hue. The functional group on the triazineylaminostilbene molecule emerged to influence on exhaustion. Usually, as the number of sulfonic groups increased on the aniline moiety, these water solubilizing groups decrease fiber substantivity as a result the exhaustion of FBs on substrate decreases and the lower level of add-on, hence the optimum whiteness obtained at higher concentrations, while compounds K5I, K5II had unsulfonated chemical structures

Table 1. The Washing Fastness of Cotton Fabric Dyed with Compounds K5I-V

Compods	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	Change of Shade
K5I	5	2-3	5	5	5	5	3
K5II	5	3-4	5	5	5	5	3-4
K5III	5	2-3	5	5	5	5	3
K5IV	5	3	5	5	5	5	3-4
K5V	5	3-4	5	5	5	5	3-4

exhibited optimum whiteness at lower concentration due to high fiber substantivity (Table 3). Compounds K5III, K5V showed optimum CIE whiteness at higher concentration (0.3% o.w.f) due to sulfonic group at R, whereas, other compounds at lower concentration, however, these compounds may favored pad application rather than the exhaustion application.

2.2. Whitening

Whitening of cotton was carried out precisely at 60°C for 30 min with a paste of finely powdered fluorescent whitening agents with a concentration of 0.05%, 0.1%, 0.2%, 0.3% and 0.5% by the weight of substrate. The liquor ratio was maintained at 1:15 and the temperature increased from 30 °C to 60 °C at rate of 2°C / min; and then for 15 min at 60 °C. The treated swatches were removed from the treatment bath, rinsed and dried in conventional manner.

2.3. Fastness

The color fastness to washing at 1/1 standard depth was assessed by using a multi-fabric specimen (tri-acetate, cotton, polyamide, polyester, poly-acrylic, and viscose) in accordance with ISO 105-CO5 as shown in Table 1. Light fastness was measured by using the Xenon arc lamp method in accordance with ISO-105-BO2, whereas the chlorine fastness test was carried out in accordance with ISO-105-EO3. The results are listed in Table 2. The comparative assessment of light fastness of compounds **K5I-V** showed fair to fairly good fastness properties.

Table 2. Lightfastness and Chlorine Fastness of Cotton Fabric Dyed with Compounds K5I-V

Compounds	Lightfastness	Chlorinated Water Fastness
K5I	4	2
K5II	3-4	1-2
K5III	4	2
K5IV	4	1-2
K5V	3-4	2-3

2.4. Whiteness Measurements

The CIE Whiteness Index (W1) value was obtained by using AATCC Test Method 110-199 [17]. The whiteness

and CIE L* a* b* coordinates were measured by using a data color spectrophotometer SF600 with the following setting: illuminant D65, large area view, specular included and CIE 1964 supplemental standard (10° Observer). Each sample was measured four times at different fabric surfaces. The average value of whiteness was recorded and data obtained are presented in Table 3.

3. EXPERIMENTAL SECTION

3.1. Materials and Analysis

¹H-NMR spectra were recorded in DMSO-d₆ by using Bruker AC (300-500 MHz) spectrometer, respectively. Splitting patterns were as follows: s (singlet), d (doublet), dd, (double doublet), t (triplet), and m (multiplet) and br (broad). Chemical shifts are reported in δ (ppm) and coupling constants are given in Hertz (Hz). The ultraviolet spectra were measured in de-ionized water on a Lambda 5 UV/Vis spectrophotometer (Perkin-Elmer). IR spectra (KBr) were recorded on a Bruker FT-IR IFS48 spectrometer. Elemental analysis was carried out on elemental analyzer-1106 (Carlo Erba). Whiteness and the CIE L*a*b* coordinates were recorded on Data Color Spectrophotometer SF-600. Light fastness results were obtained on XENONTEST 150S. The progress of all reactions was monitored by TLC, which was performed on 2.0 X 5.0 cm, PET sheets pre-coated with Silica gel with fluorescent indicator 254 nm, to a thickness of 0.25 mm. Preparative TLC analysis was carried out with Silica Gel GF 254 plates. The chromatograms were visualized under ultraviolet light (254-366 nm). The starting materials were commercially available. 4,4'-diamino-tilbene-2,2'-disulfonic acid (96%) and 2,4,6-trichloro-1,3,5-triazine (97%) were of commercial grade (Clariant, Pakistan) and all other chemicals were of analytical grade (Merck, Aldrich).

3.2. Preparation Route of Compounds

3.2.1. 5-[[4-anilino-6-(2-benzoylhydrazino)-1,3,5-triazin-2-yl]amino]-2-[(E)-2-(4-[[4-anilino-6-(2-benzoylhydrazino)-1,3,5-triazin-2-yl]amino]-2-sulfophenyl)-1-ethenyl]-1-benzenedisodiumsulfonate (K5I)

Into a 250 mL flask equipped with an agitator, pH meter, thermometer and a reflux condenser were charged with 25 ml acetone and 20 gram ice. To the stirred mixture was then added 20 mmol of 2,4,6-trichloro-1,3,5-triazine was then added to the stirred mixture. The pH was then increased

Table 3: Degree of Whiteness and Color Data for Cotton Treated with Compounds K5I-V

Compds.	o. w. f ^a	Whiteness (CIE)	L*	a*	b*
Cotton fabric	-	80.43	-0.09	0.07	-0.45
K5I	0.05	132.09	95.27	3.61	-13.02
	0.1	137.55	95.87	3.59	-13.99
	0.2	133.37	95.79	2.89	-13.07
	0.3	124.71	95.16	2.26	-11.40
	0.5	119.30	95.18	1.52	-10.18
K5II	0.05	114.43	95.19	2.57	-9.09
	0.1	114.64	94.87	2.54	-9.29
	0.2	113.90	95.21	2.48	-8.96
	0.3	113.39	95.11	2.30	-8.90
	0.5	105.04	94.63	1.80	-7.27
K5III	0.05	113.25	95.52	2.53	-8.67
	0.1	132.18	95.91	3.61	-12.76
	0.2	133.07	95.61	3.71	-13.10
	0.3	133.56	96.02	3.43	-13.01
	0.5	134.18	96.04	3.28	-13.15
K5IV	0.05	96.05	95.05	1.56	-5.27
	0.1	100.76	95.36	1.55	-5.96
	0.2	114.35	95.68	2.08	-8.84
	0.3	113.73	95.73	2.04	-8.67
	0.5	113.50	95.69	1.98	-8.64
K5V	0.05	117.38	94.85	2.93	-9.91
	0.1	128.71	94.36	3.16	-12.64
	0.2	134.78	94.99	3.47	-13.75
	0.3	134.80	95.77	3.50	-13.00
	0.5	131.94	95.83	3.13	-12.73

a: o. w. f (%) = percent concentration of the FBs on the total weight of fiber.

from about 3 to 4.5-5.5 by drop-wise addition of aqueous 10% w/v sodium carbonate solution, 50 mL disodium salt of 4,4'-diaminostilbene-2,2'-disulfonic acid solution (10 mmol) was gradually added to the slurry of cyanuric chloride while maintaining the temperature at 0-5 °C with external cooling. The reaction vessel was kept in an ice bath with continuous stirring for 1 hour while testing for the disappearance of disodium salt of 4,4' diaminostilbene-2,2-disulfonic acid by TLC analysis and iodine spot test. To the reaction mixture, 50 mL aqueous sodium salt of *p*-amino sulfonic acid (20 mmol) was added. The mixture was heated to 50 °C and stirring continued for 2 hours while maintaining pH 6-7. The (monochloro-sym-triazinyl) product from step-2 was isolated by adding 10% of sodium chloride and collected through vacuum filtration; the wet cake was used without drying in the next step. The wet cake was transferred into another flask for the next step and 150 mL of de-ionized water and 20 mmol (molar ratio was used with respect to starting material) of benzoylhydrazine was added followed by a rise in temperature up to 90 °C and stirring was continued for further 3 hours with maintaining pH at 8. The disappearance of benzoylhydrazine was monitored by TLC analysis. The reaction mixture was cooled and the product was isolated by salting-out with sodium chloride (10%). Precipitates were filtered, washed with cold (0 °C) aqueous acetone (1:1) and dried for overnight under vacuum at 40 °C. The compounds were dissolved in DMF and filtered to remove the sodium

chloride. The product was purified by preparative TLC analysis (Silica Gel GF 254 plate, amount; 25mg/1 mL); the product was homogenous as indicated by TLC analysis (Silica gel on TLC-PET foils with fluorescent indicator 254 nm, (eluent; 2-propanol, acetonitril, ammonium hydroxide (50: 20: 30)). Other FBs were prepared similarly.

C46H34N12Na2O8S2, Mol. Wt.: 992.950, Yield 86%; UV, λ_{\max}/nm , de-ionized water (352, 305, 264 nm); IR (KBr): 3296 (N-H), 1660 (C=O), 1610 (C=C, Alkene), 1598 & 1414 (C=C, Ar), 1584 (C=N), 1495 (N-H), 1364 (-CH₃), 1302 (C-N), 1182 (S=O), 898 (S-O), 988 (=C-H, bend), cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ/ppm , 6.96 (t, 2H, *J* = 7.48, 7.23 Hz, ArH), 7.13 (d, 2H, *J* = 7.78 Hz, CH=CH), 7.28 (t, 4H, *J* = 7.48, 7.36 Hz, ArH), 7.35 (d, 2H, *J* = 7.40, ArH), 7.46 (d, 2H, *J* = 7.2, ArH), 7.63 (d, 2H, *J* = 5.96 Hz, ArH), 7.72 (s, 2H, ArH), 7.80-7.87 (m, 10H, ArH), 7.98 (d, 2H, *J* = 5.35 Hz, ArH), 10.41 (br. s, 8H, NH). Elemental analysis: N% = calculated: 18.21, found: 18.08.

3.2.2. 5-((4-anilino-6-[2-(3-methylbenzoyl)hydrazino]-1,3,5-triazin-2-yl)amino)-2-((E)-2-[4-((4-anilino-6-[2-(4-methylbenzoyl)hydrazino]-1,3,5-triazin-2-yl)amino)-2-sulfophenyl]-1-ethenyl]-1-benzenedisodiumsulfonate (K5II)

C48H38N12Na2O8S2, Mol. Wt.: 1021.003, Yield 84%; UV, λ_{\max}/nm , de-ionized water (353, 306, 243); IR (KBr):

3286 (N-H), 1663 (C=O), 1596 & 1412 (C=C, Ar), 1579 (C=N), 1572 (-NH₂), 1566 (C=C, Alkene), 1411 (-CH₃), 1302 (C-N), 1182 (S=O), 897 (S-O), 791 (=C-H, bend), cm⁻¹; ¹H NMR (500 MHz, DMSO- d₆): δ/ppm 2.36 (s, 6H, CH₃), 7.27 (d, 2H, *J* = 8.76, -CH=CH-), 7.30-7.44 (m, 10H, ArH), 7.61 (d, 4H, *J* = 8.6 Hz, ArH), 7.73 (s, 2H, ArH), 7.80 (d, 2H, *J* = 3.34, ArH), 7.84 (d, 2H, *J* = 3.32 Hz, ArH), 7.88 (d, 4H, *J* = 6.5 Hz, ArH), 9.29 (s, 4H, NH), 10.37 (s, 4H, NH). Elemental analysis: N% = calculated: 17.85, found: 17.72.

3.2.3. 5-[[4-(2-benzoylhydrazino)-6-(3-sulfoanilino)-1,3,5-triazin-2-yl]amino]-2-[(E)-2-(4-[[4-(2-benzoylhydrazino)-6-(3-sulfoanilino)-1,3,5-triazin-2-yl]amino]-2-sulphophenyl)-1-ethenyl]-1-benzenedisodiumsulfonate (K5III)

C45H31N12Na4O14S4, Mol. Wt.: 1184.023, Yield 78%; UV, λ_{max}/nm, de-ionized water (353, 308, 266); IR (KBr): 3394 (N-H), 1669 (C=O), 1594 & 1412 (C=C, Ar), 1583 (C=N), 1492 (-NH), 1566 (C=C, Alkene), 1411 (-CH₃), 1309 (C-N), 1180 (S=O), 891 (S-O), 787 (=C-H, bend), cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ/ppm, 7.24 (d, 2H, *J* = 8.34 Hz, CH=CH), 7.27-7.36 (m, 6H, ArH), 7.49-7.60 (m, 8H, ArH), 7.66 (d, 2H, *J* = 7.64 Hz, ArH), 7.27 (s, 2H, ArH), 7.77 (d, 4H, *J* = 5.55 Hz, ArH), 7.90 (d, 2H, *J* = 7.66 Hz ArH), 9.47 (s, 6H, NH), 10.44 (s, 2H, NH). Elemental analysis: N% = calculated: 18.00, found: 17.24.

3.2.4. 5-[[4-(2-benzoylhydrazino)-6-(4-toluidino)-1,3,5-triazin-2-yl]amino]-2-[(E)-2-(4-[[4-(2-benzoylhydrazino)-6-(4-toluidino)-1,3,5-triazin-2-yl]amino]-2-sulphophenyl)-1-ethenyl]-1-benzenedisodiumsulfonate (K5IV)

C48H38N12Na2O8S2, Mol. Wt.: 1021.003, Yield 81%; UV (352, 309, 268); I.R (KBr): 3398 (N-H), 1673 (C=O), 1598 & 1412 (C=C, Ar), 1583 (C=N), 1492 (-NH) 1566 (C=C, Alkene), 1411 (CH₃), 1305 (C-N), 1188 (S=O), 896 (S-O), 808 (=C-H, bend), cm⁻¹; ¹H NMR(400 MHz, DMSO d₆): δ/ppm, 2.48 (s, 6H, CH₃), 7.15 (d, 2H, *J* = 7.76 Hz, CH=CH), 7.30-7.44 (m, 8H, ArH), 7.60 (d, 2H, *J* = 5.98 Hz, ArH), 7.70 (d, 2H, *J* = 5.86, ArH), 7.73-7.84 (m, 10H, ArH), 7.91 (s, 2H, *J* = 5.37 Hz, ArH), 10.42 (br. s, 8H, NH). Elemental analysis: N% = calculated: 17.87, found: 17.33.

3.2.5. 5-[[4-[2-(3-methylbenzoyl) hydrazino]-6-(3-sulfoanilino)-1,3,5-triazin-2-yl]amino]-2-[(E)-2-(4-[[4-[2-(3-methylbenzoyl)hydrazino]-6-(3-sulfoanilino)-1,3,5-triazin-2-yl]amino]-2-sulphophenyl)-1-ethenyl]-1-benzenedisodiumsulfonate (K5V)

C48H36N12Na4O14S4, Mol. Wt.: 1225.095, Yield 87%; UV, λ_{max}/nm, de-ionized water (353, 308, 267) IR (KBr): 3425 (N-H), 1668 (C=O), 1592 & 1412 (C=C, Ar), 1587 (C=N), 1572 (-NH₂) 1566 (C=C, Alkene), 1411 (-CH₃), 1305 (C-N), 1184 (S=O), 887 (S-O), 791 (=C-H, bend), cm⁻¹; ¹H NMR (400 MHz, DMSO-d₆): δ/ppm, 2.38 (s, 6H, CH₃), 7.15 (d, 2H, *J* = 7.68 Hz, CH=CH), 7.24-7.50 (m, 6H, ArH), 7.60 (d, 4H, *J* = 7.63 Hz ArH), 7.67 (d, 2H, *J* = 5.64 Hz, ArH), 7.71 (s, 4H, ArH), 7.82 (d, 4H, *J* = 5.67 Hz, ArH), 8.02 (d, 2H, *J* = 7.68 Hz ArH), 9.38 (s, 6H, NH), 10.36 (s, 2H, NH). Elemental analysis: N% = calculated: 18.43, found: 18.12.

4. CONCLUSIONS

Disodium 4,4'-bis-(1,3,5-triazinylamino)stilbene-2,2'-disulfonate derivatives were synthesized by reacting 4,4'-diaminostilbene-2,2'-disulfonic acid with 2,4,6-trichloro-1,3,5-triazine. They are suitable for the whitening of cotton fiber. These compounds provided a good degree of whiteness. As a result of this investigation, it can be assumed that the compounds (**K5I-V**) showed a high degree of whiteness and good wash fastness for cotton. The lightfastness of compounds **K5I-V** showed relatively good fastness properties. The chlorine fastness of the compound **K5II** and **K5IV** are poor ratings; it's considering that the hydrazine groups are unstable towards sodium hypochlorite.

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