

Synthesis of Some New 2-[(2,3-dihydroinden-1-ylidene)hydrazinyl]-4-Methylthiazole Derivatives for Simultaneous Dyeing and Finishing for UV Protective Cotton Fabrics

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Received 9 July 2008; accepted 12 November 2008

DOI 10.1002/app.29768

Published online 13 February 2009 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Several 2-[1-(1,2-dihydroinden-3-ylidene)hydrazono]-5-aryldiazo-4-methyl-1,3-thiazoles were synthesized by reaction of 1-(1,2-Dihydroinden-3-ylidene) thiosemicarbazide with different hydrazonyl chlorides. The products are water insoluble and UV absorbers, expressed UPF-rating values, and their H₂O/DMF solutions were used in simultaneous dyeing and resin finishing of cotton fabrics. Results obtained show that finishing of cotton samples in presence of any of that dyes, irrespective of dye concentration, brings about an improvement in percent nitrogen, wrinkle recovery angle (WRA), dyeability, and UV protection rating values along with slight decrease of tensile strength (TS) compared with the untreated sam-

ples. Irrespective of dye structure, increasing the dye concentration from 0.5 and up to 1.7% results in an improvement in the percent nitrogen, TS and a remarkable improvement in both the dyeability, UPF-rating values along with slight decrease in WRA and lower fastness properties of the treated fabrics. The treated fabrics was characterized using energy dispersive X-ray analysis indicating the entrapped dye within the fabric structure. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 112: 2221–2228, 2009

Key words: 1-indanone; thiosemicarbazone; azodyes; finishing of cotton fabrics; UV radiation

INTRODUCTION

UV radiation is a segment of the electromagnetic spectrum with a wavelength ranging from 100 to 400 nm. The prolonged exposure to UV of the wavelength ranging from 320 to 400 nm in sunlight causes loss of skin elasticity, appearance of wrinkles, promotion of the onset of erythema reaction, and the inducement of phototoxic or photoallergic reactions. Some of the more distressing embodiments of skin damage caused by excessive unprotected exposure to sunlight are development of melanomas or carcinomas on the skin.¹ Sun cream as a UV absorber protects skin against strong solar radiation. To provide clothing surrounding the skin and textile sun protective articles such as marquee or sunshades are used as additional protection against UV radiation. Most of the natural and synthetic textile fabrics, whether un-dyed or dyed, are usually at least partially permeable to UV radiation, therefore, textile fabrics coated with UV absorbers must be

used.² Protection offered by clothes is expressed by means of ultraviolet protection factor UPF.^{3,4} The ultraviolet protection factor expresses the multiple of time period for which an individual wearing the textiles (or using the suntan lotion) can spend in the sun without suffering from erythema as compared with the time period spent in the sun with unprotected skin.⁵

On the other hand, Azo dyes are the largest group of organic dyes as they constitute more than 35% of the global production of all dyes.⁶ Azo dyes have been attracting intensive interest for their potential use in optical data storage,⁷ optical switching,⁸ polarization holography,^{9,10} optical modulation,¹¹ nonlinear optics,¹² and photolabile surfactants.¹³ Moreover, many thiazole ring systems are of considerable importance because of their antibacterial and anti-inflammatory activity.^{14,15} Because of the wide spectrum of activity shown by the thiazole moiety, a large number of thiazoles substituted with different groups at various positions have been prepared.^{16–19}

Moreover, many attempts^{20–26} have been done for effecting combined dyeing and easy-care finishing of cotton fabric with a view of saving energy, time, and manpower and minimizing water pollution.

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The present work is undertaken to synthesize the first reported moiety of 2-[1-(1,2-dihydroinden-3-ylidene) hydrazono]-5-aryldiazo-4-methyl-1,3-thiazoles dyes from 1-(1,2-dihydroinden-3-ylidene) thiosemicarbazide and to use the synthesized dyes for enhancing UV protection properties of cotton-containing fabrics via simultaneous dyeing and resin finishing.

EXPERIMENTAL

Materials

Mill-scoured and bleached woven cotton fabric (125 g/m²) was used throughout this work. 1-Indanone, thiosemicarbazide, and triethyl amine were purchased from Aldrich Chemical Co. Ethanol and *N,N'*-Dimethylformamide (DMF) were purchased from EI-Nasr Pharmaceutical and Chemical Co. [(ADWIC) Egypt]. Fixapret®ECO as a finishing agent (low-formaldehyde cross-linker, based on dimethylol-dihydroxyethylene urea, BASF). Ammonium chloride and glacial acetic acid were of reagent grade.

Methods

Synthesis of 1-(1,2-Dihydroinden-3-ylidene)thiosemicarbazide (1)

Equimolar quantities (0.05 mol) of 2,3-dihydroinden-1-one and thiosemicarbazide were dissolved in 50 mL of warm ethanol, then add 3 mL of glacial acetic acid and heated under reflux for 6 h. Left to cool overnight, the crystalline product was separated by filtration, vacuum dried, and recrystallized from ethanol.^{27,28}

Synthesis of hydrazonyl chlorides (2)

Hydrazonyl chlorides were prepared according to the procedures reported in the literature.^{29,30}

Synthesis of 2-[1-(1,2-Dihydroinden-3-ylidene)hydrazono]-5-aryldiazo-4-methyl-1,3-thiazoles

General procedures. A mixture of 1-(1,2-dihydroinden-3-ylidene)thiosemicarbazide **1** (2.05 g, 10 mmol) and appropriate hydrazonyl chloride **2** (10 mmol) in ethanol containing few drops of triethyl amine was refluxed for 1.5 h. Left to cool at room temperature, the products were separated by filtration, dried, and recrystallized from ethanol/DMF.

4-((2-(2-(2,3-Dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazol-5-yl)diazenyl)benzenesulfonamide (3)

M.p. 302°C, yield (75%). UV (CHCl₃): λ_{max} = 375 nm. IR (KBr): 1596 (C=N), 3165, 3205 (NH₂); ¹H-

NMR (DMSO-d₆): 1.1 (m, 2H, CH₂); 2.45 (s, 3H, CH₃); 2.8 (m, 2H, CH₂); 4.41 (s, 1H, NH); 7.2–7.78 (m, 8H, Ar-H); 10.8 (s, 2H, NH₂). Ms (*m/z*): 428[M⁺+2](8.5); 427[M⁺+1](19); 426[M⁺](100). Anal. Calcd. for C₁₉H₁₈N₆O₂S₂ (426.52): C, 53.50; H, 4.25; N, 19.70; S, 15.04. Found: C, 53.61; H, 4.36; N, 19.76; S, 15.12.

4-((2-(2-(2,3-Dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazol-5-yl)diazenyl)-N-(pyridine-2-yl)benzenesulfonamide (4)

M.p. 296°C, yield (77%). UV (CHCl₃): λ_{max} = 395 nm. IR (KBr): 1596 (C=N), 3217 (NH); ¹H-NMR (DMSO-d₆): 1.12 (m, 2H, CH₂); 2.44 (s, 3H, CH₃); 2.8 (m, 2H, CH₂); 4.41 (s, 1H, NH); 6.52–8.13 (m, 12H, Ar-H); 11.12 (s, 2H, NH). Ms (*m/z*): 505[M⁺+2] (5.5); 504[M⁺+1] (12); 503[M⁺] (100). Anal. Calcd. for C₂₄H₂₁N₇O₂S₂ (503.60): C, 57.24; H, 4.20; N, 19.47; S, 12.73. Found: C, 57.29; H, 4.28; N, 19.53; S, 12.80.

4-((2-(2-(2,3-Dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazol-5-yl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide (5)

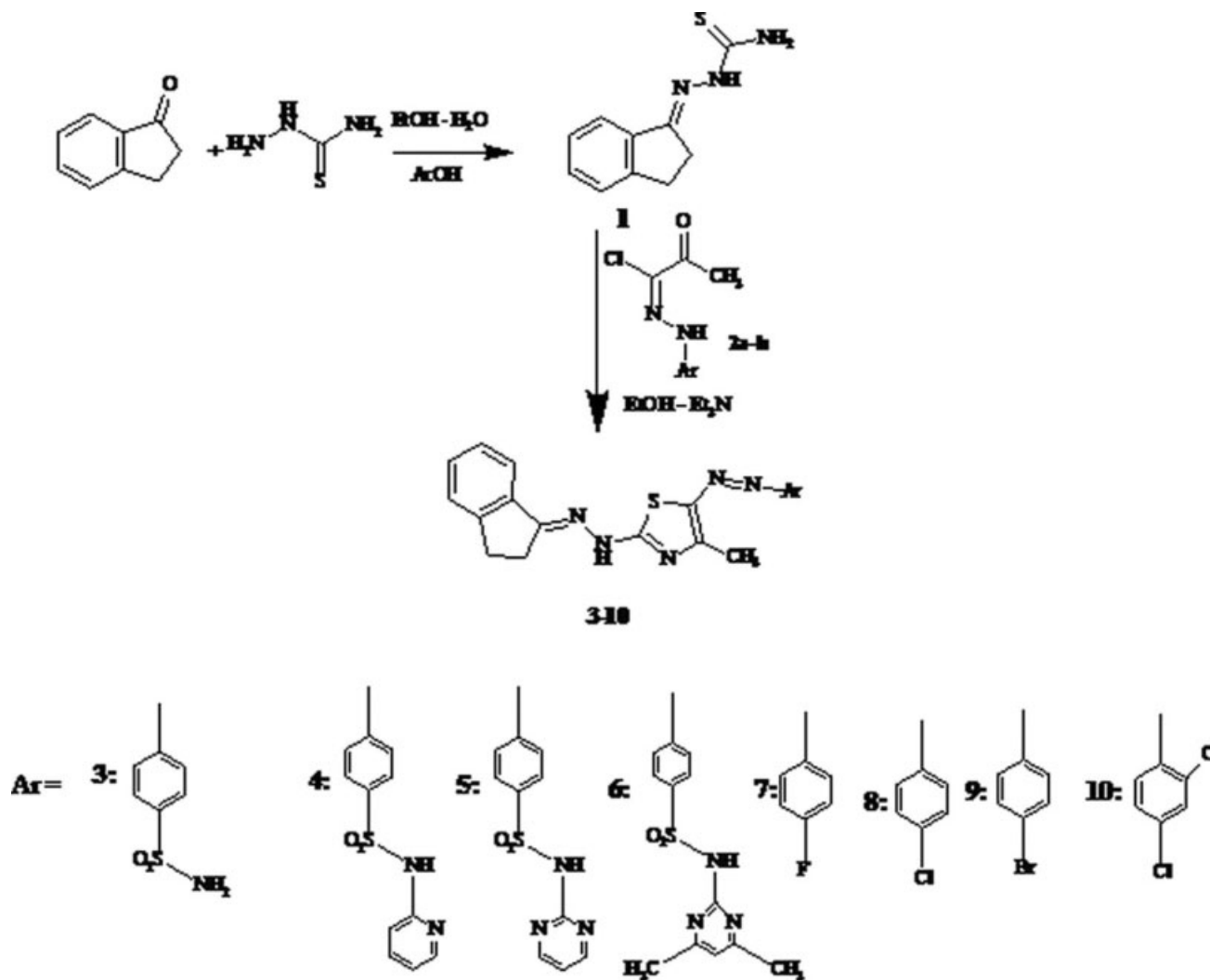
M.p. 308°C, yield (75%). UV (CHCl₃): λ_{max} = 355 nm. IR (KBr): 1591 (C=N), 3207 (NH); ¹H-NMR(DMSO-d₆): 1.11 (m, 2H, CH₂); 2.45 (s, 3H, CH₃); 2.56 (m, 2H, CH₂); 4.50 (s, 1H, NH); 6.68–8.25 (m, 11H, Ar-H); 10.88 (s, 2H, NH). Ms (*m/z*): 506[M⁺+2] (7.5); 505[M⁺+1] (17); 504[M⁺] (100). Anal. Calcd. for C₂₃H₂₀N₈O₂S₂ (504.59): C, 54.75; H, 4.00; N, 22.21; S, 12.71. Found: C, 54.81; H, 4.09; N, 22.31; S, 12.83.

4-((2-(2-(2,3-Dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazol-5-yl)diazenyl)-N-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide (6)

M.p. 220°C, yield (75%). UV (CHCl₃): λ_{max} = 460 nm. IR (KBr): 1595 (C=N), 3212 (NH); ¹H-NMR(DMSO-d₆): 1.12 (m, 2H, CH₂); 2.44 (s, 3H, CH₃); 2.36 (s, 6H, 3 CH₃) 2.53 (m, 2H, CH₂); 4.42 (s, 1H, NH); 6.32 (s, 1H, Ar-H); 6.89–7.35 (m, 8H, Ar-H); 10.93 (s, 2H, NH). Ms (*m/z*): 534[M⁺+2] (4.5); 533[M⁺+1] (13); 532[M⁺] (100). Anal. Calcd. for C₂₅H₂₄N₈O₂S₂ (532.64): C, 56.37; H, 4.54; N, 21.04; S, 12.04. Found: C, 56.43; H, 4.59; N, 21.27; S, 12.11.

2-(2-(2,3-Dihydroinden-1-ylidene)hydrazinyl)-5-((4-fluorophenyl)diazenyl)-4-methylthiazole (7)

M.p. 217°C, yield (81%). UV (CHCl₃): λ_{max} = 355 nm. IR (KBr): 1621 (C=N), 3313 (NH); ¹H-NMR (DMSO-d₆): 1.42 (m, 2H, CH₂); 2.46 (s, 3H, CH₃); 2.54 (m, 2H, CH₂); 4.40 (s, 1H, NH); 7.19–7.73 (m, 8H, Ar-H). Ms (*m/z*): 367[M⁺+2] (2); 366[M⁺+1] (12); 365[M⁺] (100). Anal. Calcd. for C₁₉H₁₆FN₅S (365.43):



Scheme 1 Structures of synthesized dyes.

C, 62.45; H, 4.41; N, 19.16; S, 8.77. Found: C, 62.51; H, 4.49; N, 19.25; S, 8.79.

5-((4-Chlorophenyl)diazenyl)-2-(2-(2,3-dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazole (8)

M.p 227°C, yield (81%). UV (CHCl₃): λ_{max} = 355 nm. IR (KBr): 1617 (C=N), 3194 (NH); ¹H-NMR (DMSO-d₆): 1.43 (m, 2H, CH₂); 2.46 (s, 3H, CH₃); 2.66 (m, 2H, CH₂); 4.54 (s, 1H, NH); 7.09–7.69 (m, 8H, Ar-H). Ms (*m/z*): 383[M⁺+2] (2.5); 382[M⁺+1] (15) 381[M⁺] (100). Anal. Calcd. for C₁₉H₁₆ClN₅S(381.88): C, 59.76; H, 4.22; N, 18.34; S, 8.40. Found: C, 59.79; H, 4.28; N, 18.47; S, 8.45.

5-((4-Bromophenyl)diazenyl)-2-(2-(2,3-dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazole (9)

M.p. 232°C, yield (81%). UV (CHCl₃): λ_{max} = 360 nm. IR (KBr): 1620 (C=N), 3208 (NH); ¹H-NMR

(DMSO-d₆): 1.41 (m, 2H, CH₂); 2.47 (s, 3H, CH₃); 2.65 (m, 2H, CH₂); 4.55 (s, 1H, NH); 7.15–7.73 (m, 8H, Ar-H). Ms (*m/z*): 427[M⁺+1] (88); 426[M⁺] (18); 425[M⁺-1] (100). Anal. Calcd. for C₁₉H₁₆BrN₅S(426.33): C, 53.53; H, 3.78; N, 16.43; S, 7.52. Found: C, 53.60; H, 3.91; N, 16.49; S, 7.62.

5-((2,4-Dichlorophenyl)diazenyl)-2-(2-(2,3-dihydroinden-1-ylidene)hydrazinyl)-4-methylthiazole (10)

M.p. 207°C, yield (79%). UV (CHCl₃): λ_{max} = 450 nm. IR (KBr): 1597 (C=N), 3208 (NH); ¹H-NMR(DMSO-d₆): 1.31 (m, 2H, CH₂); 2.47 (s, 3H, CH₃); 2.56 (m, 2H, CH₂); 4.45 (s, 1H, NH); 7.12–7.74 (m, 7H, Ar-H). Ms (*m/z*): 417[M⁺+1] (17); 416[M⁺] (8); 415[M⁺-1] (100). Anal. Calcd. for C₁₉H₁₅Cl₂N₅S(416.33): C, 54.81; H, 3.63; N, 16.82; S, 7.70. Found: C, 54.87; H, 3.69; N, 16.88; S, 7.76.

TABLE I
Effect of Dye Type and Dye Concentration of on the Extent of Resin Finishing

Dye type	Dye concentration (%)	λ_{\max}	<i>K/S</i>	%N	WRA (W+F) ^o	TS	UPF Rating
Blank	–	–	–	–	110	57	5
Control	–	–	–	0.4112	225	44.1	8
D3	0.5		4.62	0.8966	260	44.2	13
	1.7	360	5.51	0.9714	257	44.6	25
D4	0.5		3.61	0.9581	276	44.2	15
	1.7	355	5.66	0.9823	271	44.7	30
D5	0.5		4.41	1.0196	250	44.3	14
	1.7	355	5.24	1.1201	241	44.6	28
D6	0.5		3.13	0.9492	245	44.3	11
	1.7	450	5.99	1.1301	241	44.5	24
D7	0.5		5.91	0.8352	272	44.3	15
	1.7	375	7.87	0.9402	271	44.6	29
D8	0.5		3.66	0.8241	270	44.1	16
	1.7	395	6.98	0.8997	260	44.3	32
D9	0.5		5.16	0.8311	281	44.3	18
	1.7	355	6.23	0.9102	276	44.5	35
D10	0.5		5.42	0.8132	271	44.5	17
	1.7	460	6.33	0.9004	270	44.6	34

[DMDHEU], 75 g/L; [NH₄Cl], 7.5 g/L; Drying, 85°C/5 min; Curing, 160°C/3 min.

Control sample is the crosslinked sample without dyeing; all colored samples are UV absorbers in the range of 290–400 nm.

Finishing treatments

The aforementioned synthesized dyes are water insoluble, hence cotton fabric samples were padded in H₂O/DMF 40/60 solutions containing different concentrations of synthesized dyes (0.5 and 1.7%), higher concentrations make the dyes to precipitate, the finishing agent (75 g/L) and ammonium chloride as a catalyst (7.5 g/L) to give a wet pickup of 80–85% based on weight of fabric, followed by drying at 80°C for 10 min to remove the DMF² then curing at 160°C for 3 min in a circulating air oven. The finished fabrics were washed using 2 g/L nonionic detergent at 50°C/30 min, followed by washing with H₂O and finally were left for drying at room temperature.

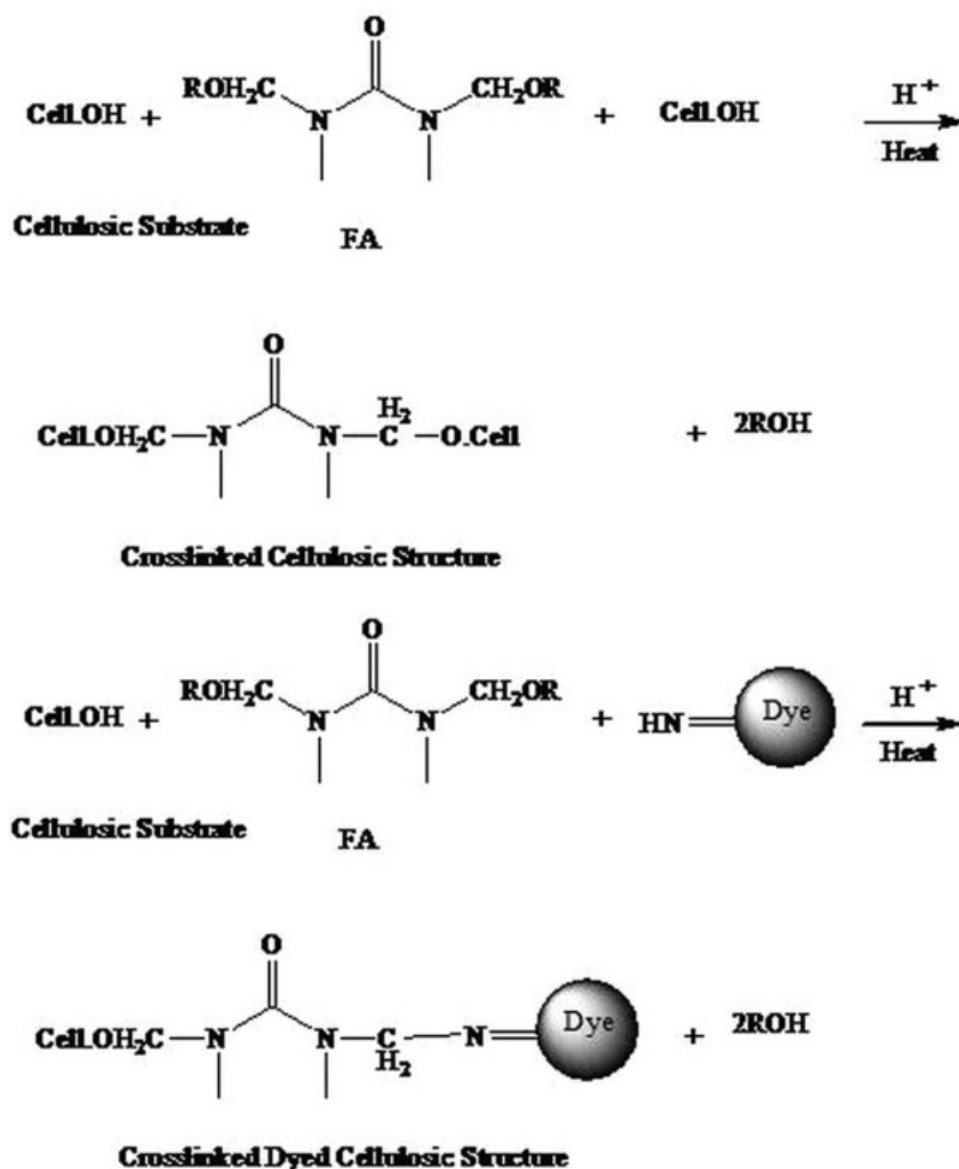
Testing methods

All melting points of synthesized dyes were taken on Electrothermal IA 9000 series digital melting point apparatus. Elemental analytical data (in accord with the calculated values) were obtained from the microanalytical unit in the National Research Center, Egypt. The IR spectra (KBr) were recorded on a Shimadzu CVT-04 spectrophotometer. The ¹H-NMR spectra were recorded at 270 MHz on a Varian EM-360 spectrometer using TMS as an internal standard. Splitting patterns are designated as follows: s, singlet; m, multiplet. Chemical shift (δ) values are given in parts per million. The mass spectra were performed using a Varian MAT CH-5 spectrometer (70 eV).

Nitrogen content (%N) of finished fabrics was determined according to the Kjeldhal method. The color strength of the of the dyed samples, expressed as *K/S* value, was measured by Optimach 3100 spectrophotometer and the values were automatically calculated from reflectance data using Kubelka-Munk equation³¹:

$$K/S = (1 - R)^2/2R$$

where *R* is the reflectance of the dyed fabric at the wavelength of maximum absorption and *K/S* is the ratio of the absorption coefficient, *K*, to the scattering coefficient, *S*. The higher the *K/S* value, the more dye uptake will be. The standard method AATCC: 66–1996 was used to evaluate conditioned wrinkle recovery angle (WRA) (W+F)^o. The tensile strength (TS) of the finished fabric sample was tested in the warp direction according to ASTM procedure D-2256-98. Fastness properties of dyed-finished fabric samples to washing (WF), rubbing (RF), and perspiration (PF) were assessed according to AATCC test methods: (91–1972), (8–1972), and (15–1973), respectively. Ultraviolet-protection factor (UPF) values were calculated according to the Australian/New Zealand standard (AS/NZS 4366-1996) with a UV-Shimadzu 3101 PC spectrophotometer. According to the Australian classification scheme, fabrics can be rated as providing good protection, very good protection, and excellent protection if their UPF values are 15–24, 25–39, and greater than 40, respectively.³² All colored samples are tested as UV absorbers in the range of 290–400 nm. The surface morphology of untreated and finished/dyed



Scheme 2 Cross-linking of the cellulose structure with resin finishing in presence of synthesized dyes. (a) Ether cross-linking.^{38,39} (b) Fixation of entrapped dyes onto the fabric matrix.⁴⁰⁻⁴²

cotton samples were examined, without further preparation, using a scanning electron microscope (JEOL, JXA-840A Electron Probe Microanalyzer, Japan) equipped with an energy dispersive X-ray system (EDX) (INCAX-Sight, England) for elemental analysis.

RESULTS AND DISCUSSION

As illustrated in Scheme 1, the key 1-indanone thiosemicarbazone (**1**) was prepared directly by reaction of 1-indanone with thiosemicarbazide, which then reacts with hydrazonyl chlorides of some primary aromatic amines and some of sulfa drugs to form the substituted 1,3-thiazoles (**3–10**).

Their ultra violet spectra in CHCl₃ showed λ_{max} at 355–460 nm, also the IR spectrum of **3** showed C=N and NH stretching bands at 1620 and 3208/cm, respectively. The mass spectra of compounds (**3–10**) are in agreement with their molecular formula.

These water insoluble synthesized products were used as UV absorbers and dyes in simultaneous dyeing and resin finishing bathes to impart UV protection to the finished fabrics and better performance properties. For simplicity, the aforementioned synthesized dyes will be designated D3–D10, according to the Ar derivative part as illustrated in Scheme 1. Results obtained along with their appropriate discussion follow.

TABLE II
Effect of Dye Type and Dye Concentration of on the Fastness Properties of Treated Cotton Fabrics

Dye type	Dye concentration (%)	Fastness properties				
		WF	RF		Perspiration	
			Dry	Wet	Acid	Alkali
D3	0.5	2	2-3	2	2	2-3
	1.7	2	2	1-2	2	2
D4	0.5	2	2-3	2	2	2-3
	1.7	2	2	1-2	2	2
D5	0.5	2	2-3	2	2	2-3
	1.7	2	2	1-2	2	2
D6	0.5	2	2-3	2	2	2-3
	1.7	2	2	1-2	2	2
D7	0.5	2-3	3	2-3	2	2
	1.7	2-3	2-3	2	2	2
D8	0.5	2-3	3	2-3	2	2
	1.7	2-3	2-3	2	2-3	2
D9	0.5	2-3	3	2-3	2	2
	1.7	2-3	2-3	2	2	2
D10	0.5	2-3	3	2-3	2	2
	1.7	2-3	2-3	2	2	2

[DMDHEU], 75 g/L; [NH₄Cl], 7.5 g/L; Drying, 85°C/5 min; Curing, 160°C/3 min.
WF, washing fastness; RF, rubbing fastness.

Simultaneous dyeing and resin finishing of cotton fabrics

Table I showed the effect of dyes structure and concentrations on the extent of resin finishing. The

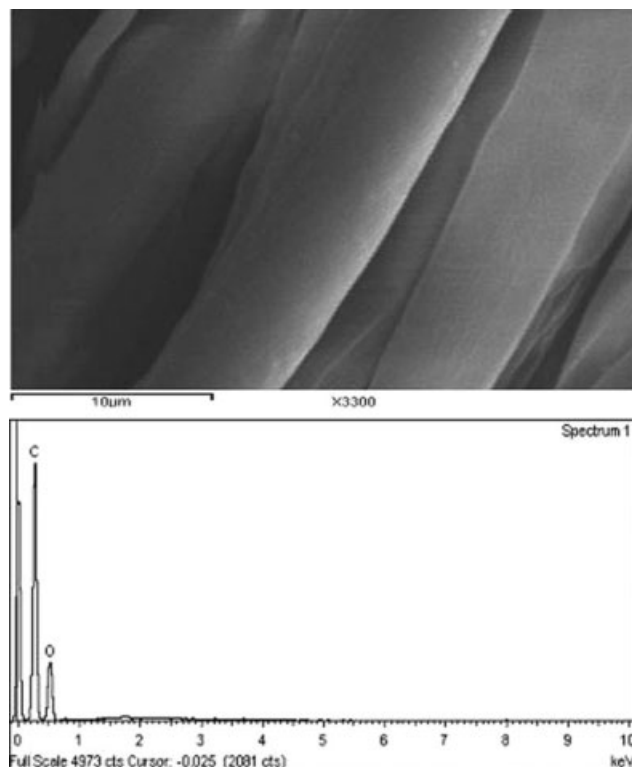


Figure 1 Scanning electron micrograph of untreated cotton fabric using energy-dispersive X-ray analysis.

results obtained signify that: (1) irrespective of dye structure, increasing the dye concentration from 0.5 and up to 1.7% brings about an improvement in the %N, TS, and a remarkable improvement in both the K/S and UPF-rating values along with slight decrease in WRA and (2) regardless of the dye concentration, the enhancement of the UV protection property is governed by the nature of the dye, that is, the chemical structure, molecular geometry, transmission/absorption characteristics, steric effect, location and aggregation, and uniformity across the fiber/fabric³³ and follows the following descending order:

$$D9 > D10 > D8 > D4 > D7 > D5 > D3 > D6$$

(3) irrespective of resin finishing, the enhancement in %N is a direct consequence of structure and concentration of the dye within cellulose structure whereas the higher fabric resiliency is attributed not only to the resin finishing but also to an additional cross-linking because of the ionic bonds between the positively and negatively charged groups present in the dyes structures.³⁴ The resiliency of the treated fabrics can be arranged in the following descending order:

$$D9 > D7 = D4 > D10 > D8 > D6 = D5 > D6 > \text{Control} > \text{Blank}$$

and (4) the extent of variation in the K/S of treated fabrics is governed by the dye molecular size,

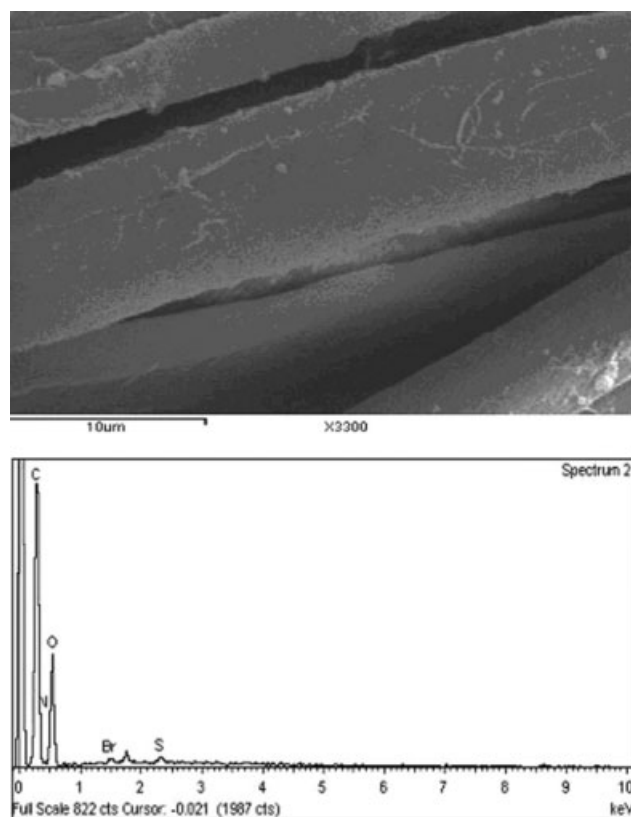


Figure 2 Scanning electron micrograph of untreated cotton fabric using energy-dispersive X-ray analysis.

functionality, reactivity, affinity, and compatibility with other ingredients, mode of interaction.^{35–37}

The presence of NH_4Cl , as a latent catalyst and the cross-linking agent in the finishing formulation along with the entrapped dyes within the fabrics matrices, would be expected to catalyze the following reactions and interactions during the curing step (Scheme 2):

Fastness properties

Table II shows the fastness properties of the treated fabrics listed in Table I. The results reveal that: (a) both of RF and WF of D3–D6 are slightly higher than that of D6–D10 reflecting the differences between such dyes in the molecular size, functionality, reactivity, affinity, and compatibility with other ingredients, mode of interaction,^{35–37} and (b) lowering the dye concentration, irrespective of the dye type, enhances the fastness properties of treated fabrics, and (c) the wet rubbing fastness properties were found to be lower than dry fastness properties, which may be associated with the presence of unfixed dye entrapped within the modified cellulose structure, irrespective of the used dye.⁴³

EDX analysis

Figures 1 and 2 show the surface morphologies and the elemental analysis of both the untreated and D9-treated cotton fabrics, respectively. It is clear that in addition to the elements of carbon and oxygen, detected in both the elemental analysis of the untreated and treated samples, the elements of nitrogen, sulfur, and bromine were also detected in only that of the treated sample indicating the entrapped dye within the fabric structure.

CONCLUSIONS

It can be concluded that 2-[1-(1,2-dihydroinden-3-ylidene) hydrazono]-5-aryldiazo-4-methyl-1,3-thiazoles are UV absorbers dyes and the simultaneous dyeing and finishing of cotton samples using any of these synthesized dyes, irrespective of dye concentration, brings about an improvement in %N, WRA, K/S, and UPF rating values along with slight decrease of TS of the treated fabrics compared with the untreated samples. Irrespective of dye structure, increasing the dye concentration from 0.5 and up to 1.7% results in an improvement in the %N, TS, and a remarkable improvement in both the K/S and UPF-rating values along with slight decrease in WRA and lower fastness properties of the treated fabrics.

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