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ORIGINAL ARTICLES

Enhancing Functional Properties of Viscose Fabric by Using Novel Aryl-Azo Sulfonamide Derivatives

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ABSTRACT

Four new sulfonamide Ary-azo dderivatives were synthesized, characterized and utilized in simultaneous dyeing and resin finishing of viscose fabrics for enhancing their functional and dyeing properties. The results indicated that these derivatives, irrespective of their concentration, brings about an improvement in the percent nitrogen, wrinkle recovery angle, homogeneous dyeability and UV protection rating values as well as antibacterial properties along with slight decrease of tensile strength of the treated fabrics. It has been also found that, irrespective of dye structure, increasing the dye concentration from 1.0 and up to 1.9% enhances the aforementioned properties accompanied with slight decrease in wrinkle recovery angle and lower fastness properties of the dyed fabrics. Moreover, the treated fabrics were characterized through investigating their energy dispersive X-ray analysis.

Key words: Sulfonamides; viscose fabrics; UV protection; antibacterial properties.

Introduction

Textiles, especially those made of natural fibers, are recognized as media to enhance the growth of microorganisms such as bacteria and fungi (Fouda *et al.*, 2013) (El-Shafei *et al.*, 2008; Fouda *et al.*, 2009; Hebeish *et al*., 2011; Hebeish *et al*., 2011). These microorganisms can be multiplied quickly where the suitable environments such as moisture, nutrients and temperature are present (Fahmy *et al.*, 2009). The growth of microorganisms on textiles causes miserable effects for the textile itself and also the wearer such as the generation of unpleasant odor, stains and discoloration in the fabric, a reduction in fabric mechanical strength and an increased like lihood of contamination. Several major classes of antimicrobial agents are used in the textile industry such as metals and metal - Salts, quaternary ammonium compounds, triclosan, chitosan, regenerable N-halamine compounds and peroxyacid and dyes (Purwar and Joshi, 2004). Azo dyes are very important compounds because of their versatile application in various fields, such as the dyeing of textile fibers and the coloring of different materials, in the food colorant, cosmetic, printing and pharmaceutical industries, in colored plastics and polymers, in biological–medical studies and for advanced applications in organic synthesis (Zollinger e, 2003). Moreover, sulfonamides are widely used in the treatment of infections, especially for patients intolerant to antibiotics. The vast commercial success of sulfonamide as antibacterial drugs made their chemistry of to become a major area of research (Nagaraja *et al*., 2003).

On the other hand, ultraviolet protection factor (UPF) of a fabric is a quantitative measurement of the effectiveness of the fabric to protect the human skin against ultraviolet radiation. Some parameters affecting a fabric UPF significantly such as fiber type, fabric structure (weave construction, yarn number, thread count, cover factor, etc.), color and dyeing intensity, presence of optical brightening agents, pigments or finishing products (especially UV-absorbers), washing conditions of the garments, etc (Algaba and Crews, 2004; Hilfiker, *et al.*, 1996; Pailthorpe, 1996; Reinert, *et al.*, 1997; Crews, et *al.*, 1999; Haerri, *et al.*, 2000; Srinivasan and Gatewood, 2000; Riva and Algaba, 2006). Meanwhile, the simultaneous dyeing and resin finishing of cellulosic fabrics has drawn the attention of many researchers (Ibrahim and Haggag, 1989). With a view of saving energy, time, manpower and minimizing water pollution.

The present work aims to improve the performance properties, UV-protection as well as antibacterial properties of viscose fabric through utilization of sulfonamide derivatives containing azo dyes**.** In this regards, four sulfonamide derivatives containing azo-dyes were synthesized, characterized and utilized in simultaneous dyeing and resin finishing of viscose fabrics. Further more, the treated fabrics were characterized through investigating their energy dispersive X-ray analysis.

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2. Experimental:

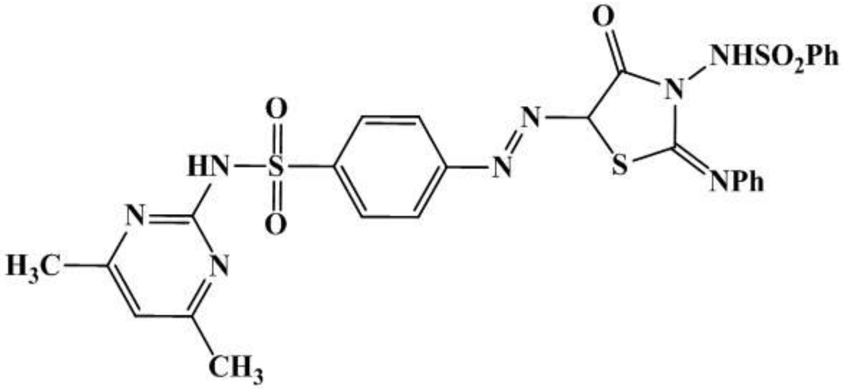
2.1. Materials:

Viscose fabric (125 g/m^2) was used throughout this work. Sulfonamide derivatives were all purchased from Sigma and were used without further purification. 2-amino-4-hydroxy-1H-pyrrole, sulfamic acid, sodium nitrite, ammonium chloride, hydrochloric acid, glacial acetic acid and all other reagents or solvents were of analytical grade. Fixapret® ECO as a finishing agent, low-formaldehyde cross-linker based on dimethyloldihydroxyethylene urea, was kindly supplied from BASF.

2.2. Methods of preparation:

The starting thiazolidin-4-one derivative was prepared according to the previous literature (Metwally *et al.*, 2004). A solution of sodium nitrite (0.70 g in 10 ml water) was gradually added to a well-cooled (0-5 ºC) solution of the sulfonamide amine (10.0 mmol) in concentrated HCl (3.0 ml) (Gaffer *et al.*, 2012).. The diazonium salt solution was added with continuous stirring to a cold (0-5 ºC) solution of thiazolidin-4-one derivative (10.0 mmol) in ethanol (50.0 ml) and sodium acetate (4.0 g). The reaction mixture was allowed to stir at (0-5 ºC) for 2 hours, and then the solid was collected by filtration. The obtained precipitate was dried and recrystallized from ethanol.The aforementioned four products are characterized and designated by D1 – D4 as follows.

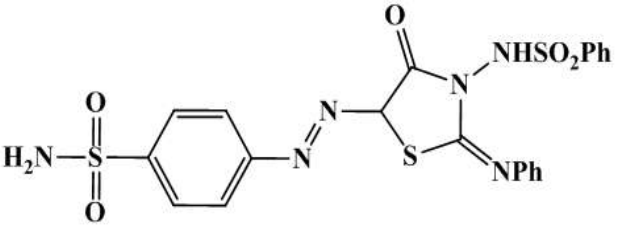
2.2.1. 3-phenylsulphonylamino-2-phenylimino-5-[**(**4-diazenyl)N-(4,6 dimethylpyrimidin-2-yl) benzenesulfonamido]-1,3-thiazolidin-4-one.



D1

M.p. 197 ^{O}C yield (67%). UV ($CHCl_3$): λmax = 478nm. IR (KBr): 1069,1079 ($2SO_2$),1515(C=C Ar),1620 (C=N), 1708 (C=O), 3312,3318 (2NH); ^{1}H NMR ($CDCl_3$) :δ/ppm = 2.35 (s,3H, CH_3), 4.45 (s, 1H, SO_2NHPH) 4.36 (s, 1H, SO_2NH),7.21-7.31(m, 15H, ArH). Ms (m/z): 638[M^+2](18.2); 637[M^+1](32); 636[M^+](100). Anal. Found: C, 50.25; H, 3.61; N, 16.46; S, 14.78. $C_{27}H_{24}N_8O_5S_3$
(636.73) requires C, 50.93; H, 3.80; N, 17.60; S, 15.11.

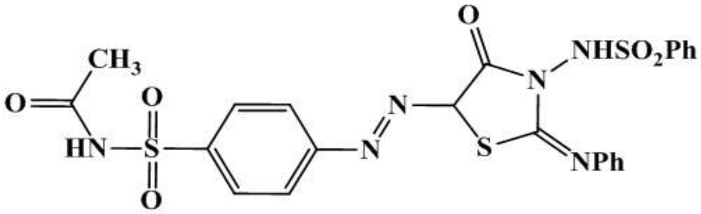
2.2.2. 3-phenylsulphonylamino-2-phenylimino-5-**[(**4-diazenyl) benzene sulfonamide]-1,3-thiazolidin-4-one.



D2

M.p. 201^{O}C yield (66%). UV ($CHCl_3$): λmax = 461nm. IR (KBr): 1075,1078 ($2SO_2$),1513(C=C Ar),1622 (C=N), 1704 (C=O), 3363,3378 (NH_2,NH); ^{1}H NMR ($CDCl_3$): δ/ppm =3.51 (s,1H,CH), 4.30 (s, 2H, NH_2), 4.00 (s,1H,NH), 7.81-7.52(m, 14H, ArH) . Ms (m/z): 532[M^+2](9.4); 531[M^+1](22); 530[M^+](100). Anal. Found: C, 46.19; H,2.71; N, 15.33; S, 17.75. $C_{21}H_{18}N_6O_5S_3$ (530.60) requires C, 47.54; H, 3.42; N, 15.84; S, 18.13.

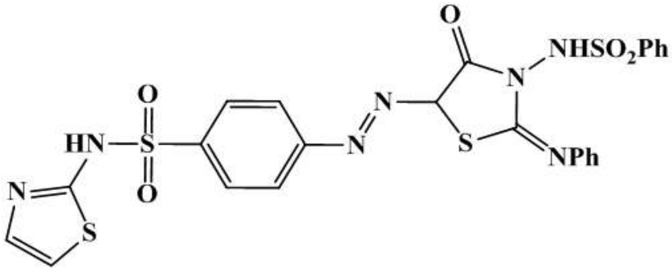
2.2.3. 3-phenylsulphonylamino-2-phenylimino-5-[**(**4-diazenyl) N-acetyl benzenesulfonamido]-1, 3-thiazolidin-4-one.



D3

M.p. 185 ^{O}C yield (73%). UV ($CHCl_3$): λmax = 473nm. IR (KBr): 1077,1079 ($2SO_2$),1508(C=C Ar),1622 (C=N),1686,1703 (2C=O), 3362,3368(2NH); ^{1}H NMR ($CDCl_3$): δ/ppm = 2.31 (s, 3H, CH_3), 3.11(s, 1H, CH), 7.72-7.66(m, 14H, ArH), 4.38-4.41 (s, 2H, NH) . Ms (m/z): 574[M^+2](4.7); 573[M^+1](18); 572[M^+](100). Anal. Found: C, 47.29; H, 3.41; N, 13.68; S, 15.84. $C_{23}H_{20}N_6O_6S_3$ (572.64) requires C, 48.24; H, 3.52; N, 14.68; S, 16.80.

2.2.4. 3-phenylsulphonylamino-2-phenylimino-5-[(4-diazenyl) N-(thiazol-2-yl) benzenesulfonamido]-1, 3-thiazolidin-4-one.



D4

M.p. 286 ^{O}C yield (68%). UV ($CHCl_3$): λmax = 476nm. IR (KBr): 1075,1079 ($2SO_2$),1510(C=C Ar),1621 (C=N),1703 (C=O), 3363,3371 (2NH); ^{1}H NMR ($CDCl_3$): δ/ppm = 3.27 (s, 1H, CH), 4.37-4.45 (s, 2H, 2NH), 7.74-7.52 (m, 16H, ArH). Ms (m/z): 574[M^+2](4.7); 573[M^+1](18); 572[M^+](100). Anal. Found: C, 46.16; H, 2.46; N, 15.28; S, 19.63. $C_{24}H_{19}N_7O_5S_4$ (613.71) requires C, 46.97; H, 3.12; N, 15.98; S, 20.90

2.3. Concurrent dyeing and resin finishing:

All the aforementioned synthesized dyes are water insoluble. As a result, the viscose fabric samples were padded in H_2O/DMF (50/50) solutions containing different concentrations of that dyes (1.0 -1.9 %), keeping in mind that the higher concentrations causes dyes precipitation, the finishing agent DMDHEU (75 g/L) and ammonium chloride as a catalyst (7.5 g/L) followed by squeezing to a wet pickup of 85% then drying at 80 ^{O}C for 10 min to remove the DMF followed by curing at 160 ^{O}C for 3 min in circulating air oven. The finished fabrics were washed using 2 g/L nonionic detergent at 50 ^{O}C /30 min, followed by washing with H_2O and finally were left for drying at room temperature.

2.4. Testing methods:

Melting points of synthesized dyes were measured using 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 (d) 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 (Algaba *et al.*, 2004). The color strength of the of the dyed samples, expressed as K/S value, was measured by Optimacth 3100 spectrophotometer and the values were automatically calculated from reflectance data using Kubelka–Munk equation (Blanchard *et al.*, 1991).

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

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 (Srinivasan and Gatewood, 2000). All colored samples are tested as UV absorbers in the range of 200-800 nm. Antimicrobial activity against Gram-positive bacteria (*Staphylococcus Aureus*, SA) and Gram-negative bacteria (*Escherichia Coli*, EC) was tested according to AATCC Test Method 100-1999. The surface morphology of untreated and finished/dyed 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

The aforementioned azo dyes, D1-D4, were synthesized in order to be utilized in the concurrent dyeing and resin finishing of viscose fabrics to enhance their UV protection as well as antibacterial properties. Results obtained along with their appropriate discussion follow.

3.1. Simultaneous dyeing and resin finishing of viscose fabrics:

Table 1 shows the effect of dyes structure as well as concentration on the extent of resin finishing. It is clear that, irrespective of dye structure, increasing the dye concentration from 1.0 to 1.9 % results in: 1) an enhancement in the %N which is a direct consequence to the presence of the nitrogen containing dye structure within cellulose structure, 2) slight improvement in TS and a remarkable improvement in the *K/S* along with slight decrease in WRA which can be associated with the accompanying increase in the finishing bath viscosity, which hinders the diffusion of DMDHEU inside the fabric structure 1, and 3) the extent of variation in the K/S of treated fabrics is governed by the dye molecular size, functionality, reactivity, affinity, as well as compatibility with other ingredients, mode of interaction (Ibrahim *et al.*,1996; Blanchard *et al.*, 1992; Shore, 1991).

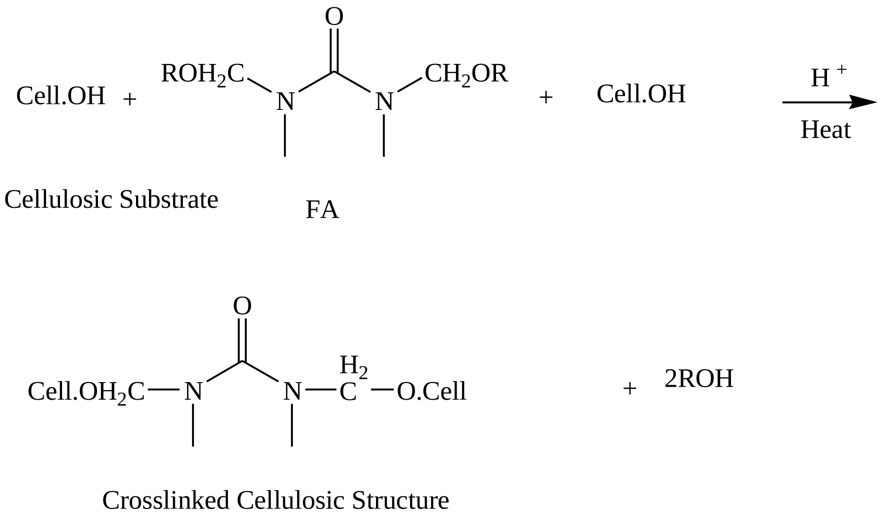
Table 1: Effect of the dye type and concentration on the extent of resin finishing.

Dye Type	Dye Conc.(%)	λ_{max}	K/S	%N	WRA $(W+F)^{O}$	TS
Blank	-	-	-	-	170	40
Control	-	-	-	0.3211	210	32
D1	1.0	478	26.1	0.6539	220	32.4
	1.9		28.3	0.8135	194	32.5
D2	1.0	461	27.6	0.7014	240	32.7
	1.9		29.4	1.2173	200	32.9
D3	1.0	473	28.2	0.6962	210	32.5
	1.9		29.9	0.8931	190	32.8
D4	1.0	476	25.3	0.5358	200	32.4
	1.9		26.8	0.7174	185	32.5

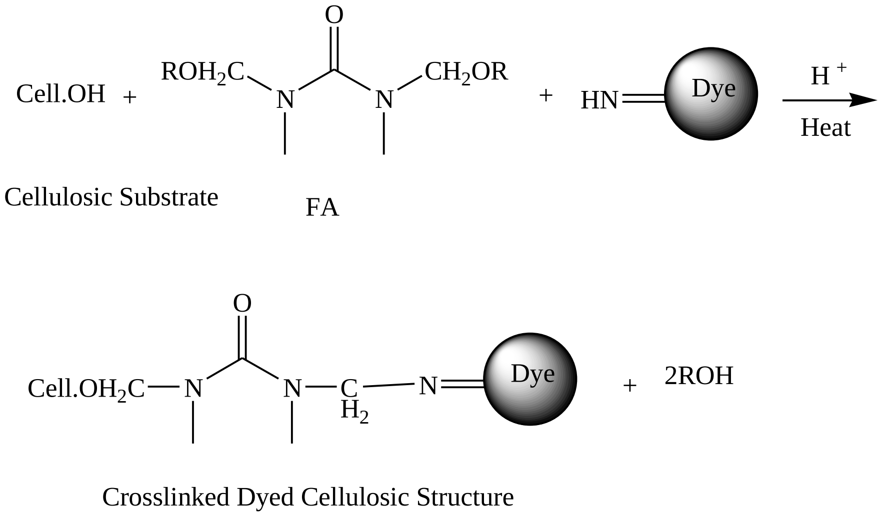
Concentration of DMDHEU, 50 g/l; concentration of ammonium chloride, 0.5 g/l; drying temperature, 85 ^{O}C/3min; curing temperature, 160 ^{O}C/3 min.

Using the NH_4Cl as a latent catalyst in the finishing formulations in the presence of the aforementioned dyes would be expected to catalyze the following reactions and interactions during the curing step:

a) Ether – crosslinking (Hebeish *et al.*, 1982; Cardamone *et al.*, 1997).



b) Fixation of entrapped dyes onto the fabric matrix (Cardamone *et al*., 2000; Cardamone *et al*., 1996; Lewis et al., 1997).



Scheme 1: Crosslinking of the cellulose structure with DMDHEU in presence of sulfonamides containing azo dyes.

3.2. Fastness properties:

Table 2 shows the fastness properties of the treated dyed viscose fabrics. It is clear that: a) irrespective of the dye concentration, the RF and WF of D2 and D3 treated samples are slightly higher than other treated samples reflecting the differences between such dyes in the molecular size, functionality, reactivity, affinity, as well as compatibility with other ingredients, mode of interaction (Ibrahim *et al*., 1996; Blanchard *et al*.,1992; Shore *et al*., 1991)., b) increasing the dye concentration, irrespective of the dye type, lowers 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 within the viscose structure, irrespective of the used dye (Ibrahim *et al*., 2005).

3.3. Antibacterial activity as well as UV protection of treated viscose fabrics:

The antimicrobial activity and UV protection of treated viscose fabrics was listed in Table 3. The results signify that, irrespective of the dye structure: 1) all treated fabrics have antibacterial properties as the used dyes are antibacterial sulfonamides derivatives (Gaffer *et al*., 2012) and increasing the dye concentration from 1.0 to 1.9 % is accompanied with increasing the bacteria inhibition zones and follows the descending order D2 > D3 > D1> D4, and 2) all treated fabrics blocks the UV radiation and increasing the dye concentration results in an improvement in the UPF-rating values and follows the descending order: D2 > D3 > D1 > D4. Irrespective of the dye concentration, the variations in both such properties may be associated with the dye nature, that is, the chemical structure, molecular geometry, transmission/absorption characteristics, sterric effect, location and aggregation, and uniformity across the fiber/fabric (Ibrahim *et al*., 2005).

Table 2: Effect of the dye type concentration on the fastness properties of treated viscose fabrics.

Dye Type	Dye Conc. (%)	Fastness Properties				
		WF	RF		Perspiration	
			Dry	Wet	Acid	Alkali
D1	1	2	2-3	2	2	2-3
	1.9	2	2	2	2	2
D2	1	2-3	3	2-3	2	2
	1.9	2-3	2-3	2	2-3	2
D3	1	2-3	3	2-3	2	2
	1.9	2-3	2-3	2	2	2
D4	1	2	2-3	2	2	2-3
	1.9	2	2	2	2	2

[DMDHEU], 75 g/l; [NH_4Cl], 7.5 g/l; drying, 85 ^{O}C / 5 min; curing, 160 ^{O}C / 3 min; WF: washing fastness; RF: rubbing fastness.

Table 3: Effect of the dye type and concentration on the UPF-rating values and antibacterial activity of treated viscose fabrics.

Dye Type	Dye Conc. (%)	UPF Rating	Inhibition Zone in mm	
			Staphylococcus aureus	Escherichia coli
Blank	-	2	-	-
D1	1	22	15	25
	1.9	35	30	34
D2	1	28	20	31
	1.9	43	36	38
D3	1	26	17	27
	1.9	37	33	34
D4	1	21	12	22
	1.9	33	24	30

[DMDHEU], 75 g/l; [NH_4Cl], 7.5 g/l; drying, 85 ^{O}C / 5 min; curing, 160 ^{O}C / 3 min.

3.4. EDX analysis:

Figures 1 and 3 show the surface morphologies whereas Figures 2 and 4 show the elemental analysis of both the untreated and D2-treated viscose fabrics, respectively. It is clear that the elements of carbon and oxygen were detected in the elemental analysis of the untreated sample whereas in addition to carbon and oxygen, the nitrogen and sulfur were also detected in the D2 treated samples indicating the presence of the D2 dye onto the fabric structure.

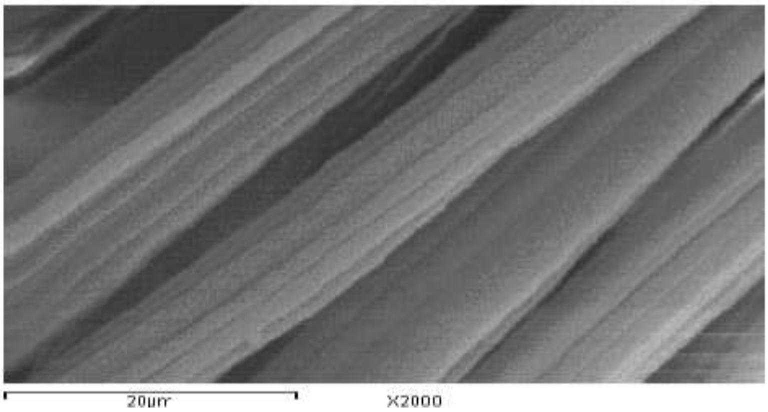


Fig. 1: Scanning electron micrograph of untreated viscose fabric.

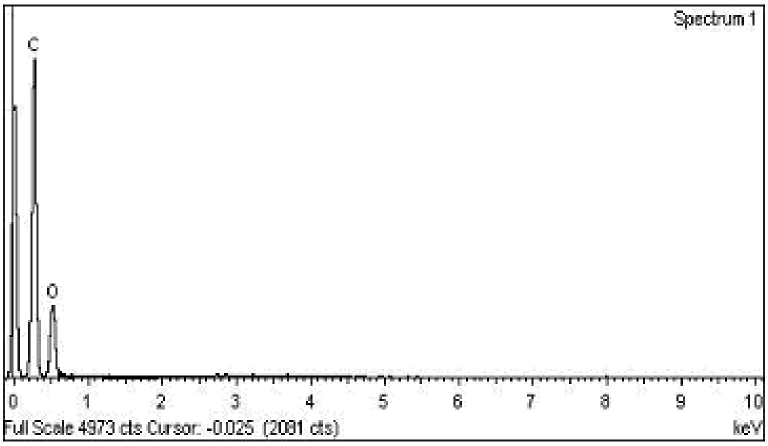


Fig. 2: Energy - dispersive X-ray analysis of untreated viscose fabric.

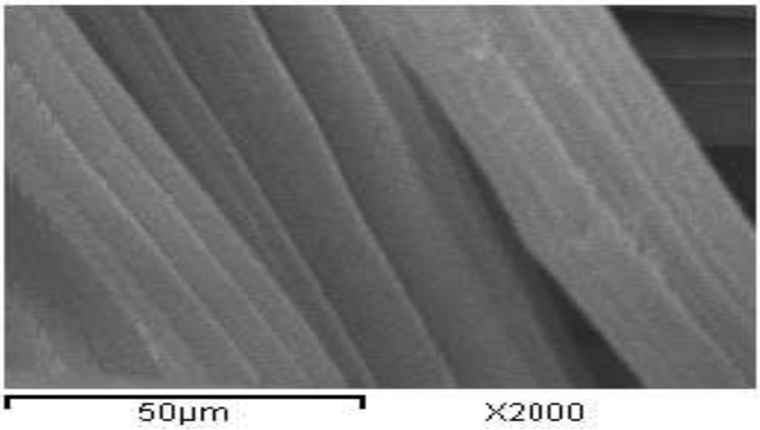


Fig. 3: Scanning electron micrograph of D2 treated viscose fabric.

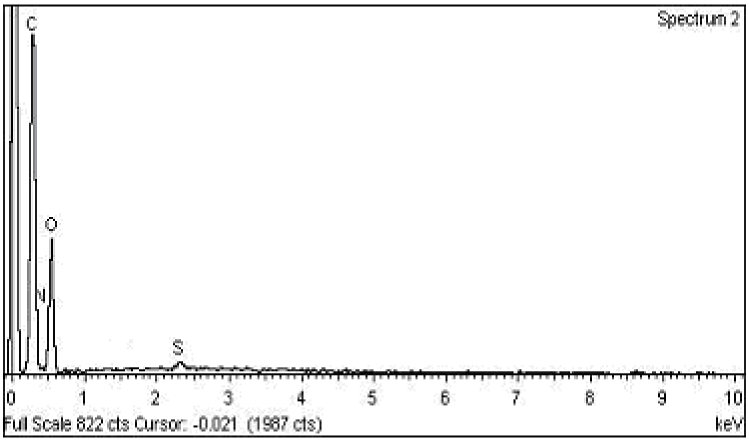


Fig. 4: Energy - dispersive X-ray analysis of D2 treated viscose fabric.

Conclusion:

Sulfonamide derivatives containing azo dyes can be utilized in simultaneous dyeing and resin finishing of viscose fabrics to enhance the performance properties, UV protection as well as antibacterial properties of treated fabrics irrespective of dye concentration. Irrespective of dye structure, increasing the dye concentration from 1.0 and up to 1.9 % enhances the aforementioned properties accompanied with lower fastness properties of the treated fabrics.

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