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BI-FUNCTIONAL REACTIVE DYES: A STUDY OF THEIR DYEING PROPERTIES ON COTTON FABRIC

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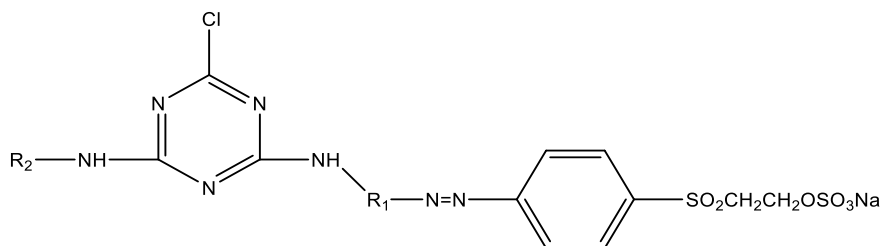
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Abstract

Bifunctional reactive dyes were examined considering new reactive dyes fixation with wet fastness properties on to cotton fiber. The bi-functional reactive dyes along various groups 4-(β -sulfatoethylsulfonyl) aniline were studied on to cotton fabric with exhaust dyeing method to investigate the performance of groups on fixation of shades, tone and wet fastness behavior. The bi-functional reactive dyes synthesized by the coupling of diazotized 4-(β -sulfatoethylsulfonyl) aniline by different cyanurated coupling components in good yield. These dyes were characterized by FTIR, ¹H NMR spectroscopic elemental analysis. Colorimetric data (L*, a*, b*, C*, H*, K/S) have been studied. The dye with various position of groups has individual affectability and affinity to the used cotton material. Synthesized bi-functional dyes performed good fixation at most favorable settings of temperature, pH and dyeing time were studied. Colorimetric data and K/S have also been investigated.

Graphical Abstract



Structure bifunctional reactive dyes

Keywords: Bi-functional Reactive dyes, Cotton, Structure of dyes, Exhaust, Fastness properties.

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1. Introduction

Cellulosic fibers can be dyed with different dyes like reactive, direct, vat and sulphur. Due to competitively greater fastness properties and simplicity of application reactive dyes are the most generally used in textiles industry. Reactive dyes are used for dyeing and printing of cotton, wool, nylon, and silk [1,2]. Commercially 1,00,000 types of dyes available, with over 7×10^5 tons of dye-stuff produced annually, dyestuff can be classified in keeping with their structure as anionic and cationic [3]. It has been observed that the fixation and colour strength of dye depends upon nature of fabric, dyebath pH, dyeing time, methodology of dyeing, auxiliaries which used in dyeing process, dyeing time and dyes synthesis behaviors [4-5]. The dyeing properties and wet-fastness of reactive dyes can be achieved with modification and introduced new molecules in dyes structure. Reactive dyes structure have two main component first is monochlorotriazine (MCT) and second is sulphatoethylsulphone (SES) displayed better fixation power compared to vinyl sulphone foregoer. During exhaust in alkali medium dyes containing only one group in structure which create bond to the cotton by meaning of nucleophilic substitution along with Michael addition [6]. Sulphatoethylsulphone (SES) models of bi-functional reactive dyes presumably assist to reduce the dye effluent loads, giving to an environmentally-developed process [7]. The dye fixation, colour strength and wet-fastness properties achieved with optimize the dyeing condition. In textiles industry the wastewater contains high concentrations of reactive dyes is a well-known problem [8]. Reactive dyes represents 25-30% of the total synthesis dye industry, therefore substantial number of research on wastewater treatment has ambition on the termination of reactive dyes [9]. Many studies have been dedicated to developing the substantivity of cotton fibers for

reactive dyes, therefore removing the quantity of electrolytes utilized [10]. In the structure of azo reactive dyes containing Azo bond $N=N$ acquired principally though the aromatic amine, Azo dyes discharged to environment toxicity it is obligatory to better and come about novel methods of application and structure improvement [11].

Consequently, to establish sustainable environmental evolution, the evacuated of synthetic waste water ought to be decreased to maximum level, that ever requirements the development of operation fluctuating dyes division [12-17]. The dye fixation of reactive dyes during dyeing affect from the dyeing technique, dyeing group, dye concentration, dyeing temperature, time of dyeing cycle, pH of dyebath and amount of alkali. Additionally, chemical group and its position in dye structure are besides significant in achieving the colour fixation [18]. Phenyl urea derivatives increase the wet fastness properties of reactive dyes [19]. In this research paper, four bi-functional reactive dyes containing functional group in different position were preferred and evaluated on cotton texture.

2. Experimental

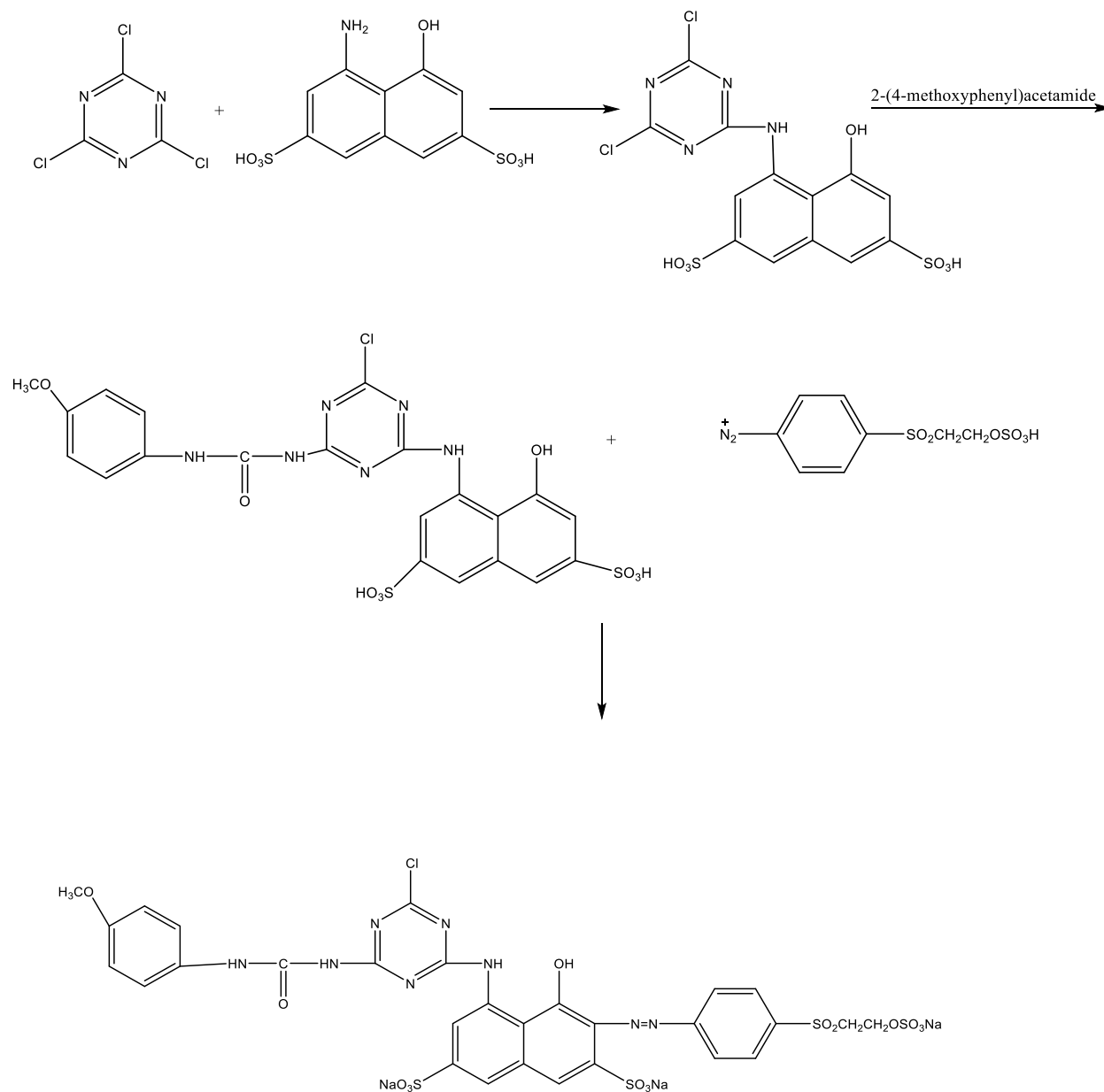
2.1 Materials

All chemical 1-amino-8-naphthol-3,6-disulphonic acid (H-acid), 4-hydroxy 7-methyl amino naphthalene-2 sulphonic acid and cyanuric chloride were obtained from Astik dyestuff. All the other chemicals involved in this study were of chemical grade. Silica gel 60₂₅₄ Aluminum sheets was used for thin layer chromatography. For exhaust dyeing bleached and knitted fabric was obtained from DyStar India Pvt Ltd. The dyes used for exhaust dyeing of cotton are specified in Figure 8, Coupling component were used are shows in Table 3. The λ_{max} of dyes were measured by SS 5100 A Premier Color scan spectrophotometer. IR dyeing machine Mfg. R.B. Electronics and Eng. Pvt. Ltd. India was used for exhaust

dyeing process of cotton fiber. Data color 400 TM spectrophotometer was used for color measurement. Na_2SO_4 , Na_2CO_3 and Nonionic detergent was used for exhaust and washing of fabric.

2.2 Synthesis of dyes

Reactive dyes D1-4 were synthesized as per described scheme 1, only change in coupling component N methyl J acid replaced with H-acid and VS meta replaced with para.



Scheme 1: Synthesis procedure of the reactive dyes

2.2.1 Preparation of cyanurated coupling compounds

Cyanuric chloride (3.19 g, 0.01 mol) was stirred in acetone below 5°C for a period of 2 hours, a neutral solution of H-acid was added in small amount in about 1 hour

the pH 4 was maintained by addition of 20% (w/v) sodium carbonate solution the reaction mass was continued stirred for 4 hours. The development of the reaction was continued by TLC using n-PrOH: n-BuOH:EtOAc:H₂O, (2:4:1:3, v/v). cyanurated H-acid is created in a clear

solution. The temperature of reaction mass was continuously increased to 45° C. 2-(4-methoxyphenyl) acetamide (1.65 gm, 0.01 mole) was introduced slowly to cyanurated H-acid over a 1 hour interval, the pH was maintained neutral by adding sodium carbonate 20% (w/v) at the same time, stirring proceeded for an further 3 hour after finished reaction, the condensation product was used for the subsequent coupling reaction.

2.2.2 Diazotization of 4-(β -sulfatoethylsufonyl) aniline

4-(β -sulfatoethylsufonyl) aniline (2.81 g, 0.01 mol) was dissolved in 15 mL H₂O at pH 6-7 by addition a sodium carbonate (10%, w/v) solution continued by cooling to 0-5°C, sodium nitrite (0.72 gm, 0.0105 mol) in H₂O add to solution of 4-(β -sulfatoethylsufonyl) aniline. The reaction mass was cooled at 0-5°C concentrated hydrochloric acid (37%, 2.5 ml, 0.015 mol) was added in this mass under vigorous stirring. For 1 hour reaction mass was continued stirring at 0-5°C with a positive test for nitrous acid. By adding a sulfamic acid the excess nitrous acid was decomposed. The reaction mass was used for the subsequent coupling reaction.

2.2.3 Coupling reaction

The diazonium salt of 4-(β -sulfatoethylsufonyl) aniline was added slowly to the condensation solution prepared previously at 5-10° C. The pH was maintained at 7-8 by adding 20% (w/v) Na₂CO₃ solution stirring was continued 1 hour. After the completion of reaction the dye was salted by using NaCl, the dye was collected by filtration and washed with NaCl (5%, w/v). The salt was removed by stirring the dye with dimethyl formamide. The collected dye was washed with EtOAc and dried at 45°C.

2.3 Dyeing methodology

Exhaust dyeing were performed with 2 gm fabric in a liquor ratio 1:20. In figure 1 is presented. For exhaust dyeing using 2% of dye solution. Different setting for the multiplication of the exhaust conditions were examined i.e., temperature (50°,60° and 70° C), dyeing time 50,60 and 70 min.) and pH (9,10 and 11). Solution of Na₂SO₄ (40 g L⁻¹) and Na₂CO₃ (20 g L⁻¹) were added (figure 1). Dye, Salt and Na₂CO₃ were calculated on the basis of fabric [20].

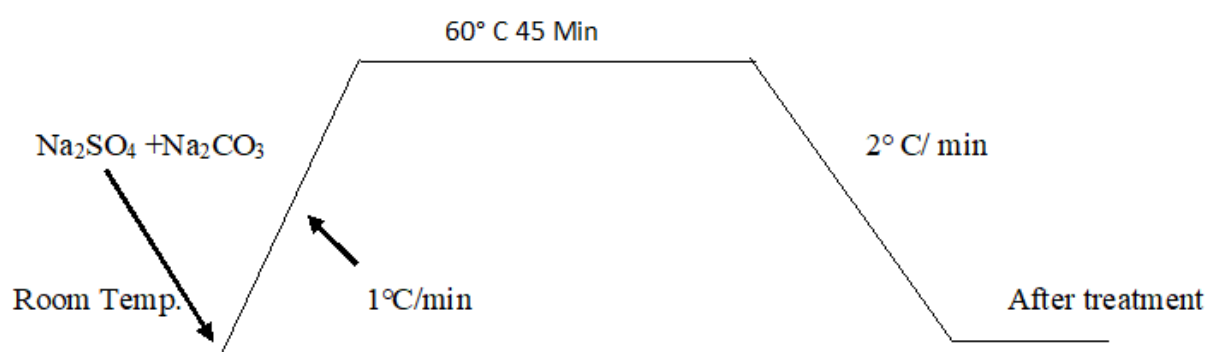


Figure 1: Dyeing curved for cotton fabric dyeing

The dyed cotton fabric after completion of exhaust was rinsed in cold water and then rinsed in warm water for 10 min. and then. Soaped-off using 1 g L⁻¹ non-ionic washing agent to remove the unfixed reactive dye. After soaping the dyed fabric was then dried at room temperature. The exhaustion and fixation values were evaluated using spectrophotometer by equation 1 and 2. [21-23].

$$\% \text{ Exhaustion } E = (A_o - A_t) / A_o \times 100\% \quad (1)$$

$$\% \text{ Fixation } F = (A_o - A_t - A_s) / A_o \times 100\% \quad (2)$$

A_o is the absorbance of initial dye bath, A_t is residue dye bath after dyeing and A_s is the absorbance of the soaping liquor.

ISO methods used for colour fastness testing i.e., washing fastness tests carried

out according to ISO 105-CO2, light fastness was determined according to ISO 105-B02(1988), rubbing fastness carried out according to ISO 105-X12 (1987) were used [24-25].

3. Results and discussion

3.1 Spectral data of dyes

The structure of four synthesized dyes were confirmed by IR and ¹H NMR (400 MHz) in DMSO d₆ as a solvent. The IR spectra of dyes (Table 1) showed characteristic broad absorption band at 3443-3446 cm⁻¹ corresponding to the O-H, N-H stretching vibration of -OH and secondary amine. C-H stretching vibration of -CH₂ group at 2929-2930 cm⁻¹, C=O strength vibration at 1675-1680 cm⁻¹, N=N strength vibration of azo group at 1564 cm⁻¹, Triazine ring vibration at 1485 cm⁻¹.

Table 1: IR spectra data of reactive dyes D1-D4

Dye No.	IR Cm ⁻¹
D1	IR (KBr, cm ⁻¹): 3443 (N-H, O-H), 2930 (C-H), 1675 (CO), 1594 (N-H), 1564 (N=N) 1485 (triazine), 1385, 1241, 1034 (-SO ₃ H), 1030,1267 (C-O-C).
D2	IR (KBr, cm ⁻¹): 3442 (N-H, O-H), 2933 (C-H), 1678 (CO), 1598 (N-H), 1564 (N=N) 1485 (triazine), 1385, 1246, 1034 (-SO ₃ H), 1030,1266 (C-O-C).
D3	IR (KBr, cm ⁻¹): 3444 (N-H, O-H), 2932 (C-H), 1675 (CO), 1594 (N-H), 1564 (N=N) 1485 (triazine), 1385, 1250, 1034 (-SO ₃ H), 1030,1267 (C-O-C).
D4	IR (KBr, cm ⁻¹): 3446 (N-H, O-H), 2929 (C-H), 1680 (CO), 1596 (N-H), 1564 (N=N) 1485 (triazine), 1385, 1245, 1034 (-SO ₃ H), 1030,1268 (C-O-C).

¹H-NMR spectra of dyes (Table 2) showed singlet at 3.15-3.25 ppm of -OCH₃, singlet at 5.70-5.71 ppm of -CH₂- protons, multiplet at 6.40-7.68 ppm of Ar-H proton, singlet at 9.32-9.45 ppm of -OH protons and multiplet at 9.31-9.71 ppm of -NH-.

Table 2: ^1H NMR spectral data of reactive dyes D1-4

Dye No.	^1H NMR (ppm)
D1	3.15 (s, 3H, OCH ₃), 5.70 (s, 1H, SO ₂ CH ₂); 6.40-7.64 (m, 12H, Ar-H), 8.53 (s, 1H, SO ₃ H) 9.32 (s, 1H, OH), 9.31-9.62 (m, 3H, NHCONH, NH) δ ppm
D2	3.16 (s, 3H, OCH ₃), 5.71 (s, 1H, SO ₂ CH ₂); 6.41-7.65 (m, 12H, Ar-H), 8.54 (s, 1H, SO ₃ H) 9.33 (s, 1H, OH), 9.31-9.63 (m, 3H, NHCONH, NH) δ ppm
D3	3.18 (s, 3H, OCH ₃), 5.705 (s, 1H, SO ₂ CH ₂); 6.41-7.68 (m, 12H, Ar-H), 8.58 (s, 1H, SO ₃ H) 9.38 (s, 1H, OH), 9.38-9.70 (m, 3H, NHCONH, NH) δ ppm
D4	3.25 (s, 3H, OCH ₃), 5.70 (s, 1H, SO ₂ CH ₂); 6.40-7.64 (m, 12H, Ar-H), 8.59 (s, 1H, SO ₃ H) 9.45 (s, 1H, OH), 9.39-9.71 (m, 3H, NHCONH, NH) δ ppm

3.2 Effect of temperature

Temperature of dyeing affects chemical properties, temperature effect was examined in the ranges of 50^o, 60^o and 70^o C, although further parameter were continual and returned disclosed such temperature concerned the dyes exhaustion and fixation accordingly (Fig.2 ,3). Dye D2 and D4 indicate maximum dyeing and fixation value at 60^o C this dyes have same (meta base 4-(β -sulfatoethylsulfonyl) aniline) groups present in dyes structure, although dye D1 and D3 provided lower strength at 60^o C dyes have para base group in structure. The exhaustion and fixation values of dyes high at 60^oC in dyes D2 and D4. The dyes having comparable functional group (same meta position) gave better fixation value, and as the functional group (para 4-(β -sulfatoethylsulfonyl) aniline) changed the reactivity of dyes also changed. In bifunctional reactive dyes with two reactive systems, therefore the triazin and vinylsulphone structure system are given better levels of dye fixation.

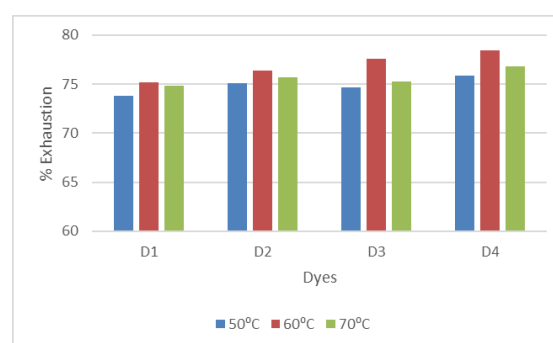


Figure 2: Effect of dyeing temperature (50-70^oC) on % exhaustion of four reactive dyes

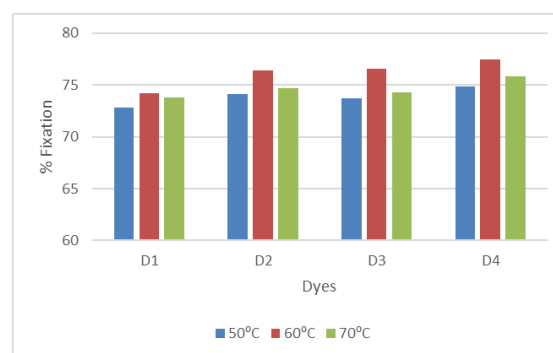


Figure 3: Effect of dyeing temperature (50-70^oC) on % fixation of four reactive dyes

At 70^oC temperature hydrolytic debasement of dye can appear in aqueous medium, while 50^oC gathering of dye fragment and avoidance of dyes to fix on fiber [26,27]. To achieve superior results 60^oC dyeing temperature was considered as acceptable temperature for following dyeing procedure.

3.3 Effect of pH

In reactive dyeing necessary to add alkali for generate hydroxyl ions on cellulose fiber to form vinylsulphone group from sulphato ethylsulphone reactive group [28]. During exhaust dyeing cellulosic dyes are heavier perceptible to pH, although pH of the dye bath liquor is first of important parameter in the application process. The group of hydroxyl in 8 to 12 pH interface besides electron weak carbon affixed to the group of chloro and maximum temperature needed to relieve the third group and to create covalent bond. Comparably, reactive dyes obtain activated under alkaline pH at maximum temperature to exhibit the reactive form of vinylsulphone, that collaborate with O-nucleophile in the cellulosic fabrics and consequently, a covalent linkage constituted along Michael addition mechanism [29-30].

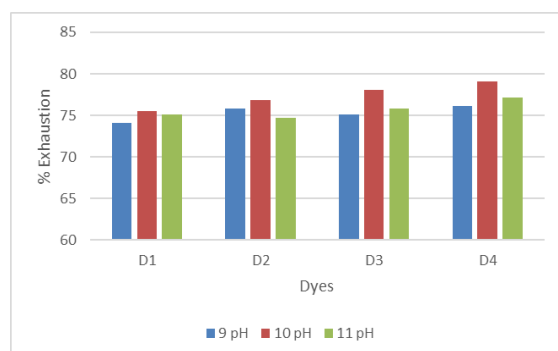


Figure 4: Effect of pH (9-11) on % exhaustion of four reactive dyes

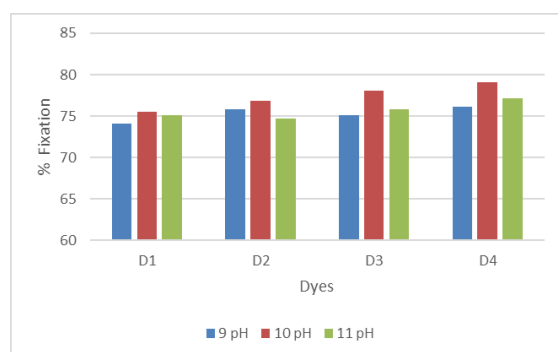


Figure 5: Effect of pH (9-11) on % fixation of four reactive dyes

The hydrolysis rate of reactive dyes minimum at 10 pH due to this reason the exhaustion and fixation percentage were high at 10 pH (Figure 4,5). The monochlorotriazine and bi-functional reactive dye have a same level of reactivity, pH of dyebath is important factor for hydrolysis of reactive dye. D1 and D3 containing para ester can be easily attacked and hydrolyzed because of to simple planar structure, all four dyes keeping equal functional groups at various situation indicated contrasting fixation value [31].

3.4 Effect of Dyeing time

The exhaust actions of synthesized dyes under examination, as an operation on time is indicate Fig. 6,7. The evaluation voiced 60 min exhaust time submitted high fixation value of D2 and D4 dyes. In the dyeing methodology of reactive dye shallow adsorption, diffusion and dye - fabric fixation factors play main roll [32]. D2-D4 showed maximum exhaustion, while D1-D3 exhaustion and fixation was least. The higher temperature of dyeing the lower substantivity of the dye resulted, and the rate of hydrolysis also increase. The rate of meta and para base 4-(β -sulphatoethylsulfonyl) aniline dyes hydrolysis effected with temperature. The capillaries of inside the fiber are more open at 60^o C temperature compared to 50^o Cand 70^o C in meta position in dyes structure. Furthermore, thermal motion of dye structure towards the fiber surface was also increased [33].

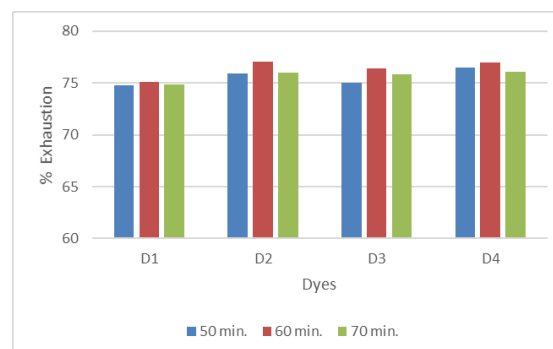


Figure 6: Effect of dyeing time (50-70 min.) on % exhaustion of four reactive dyes

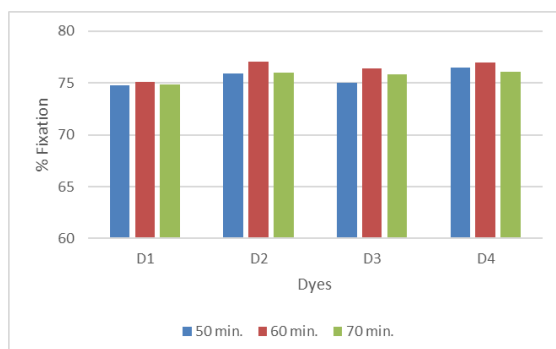


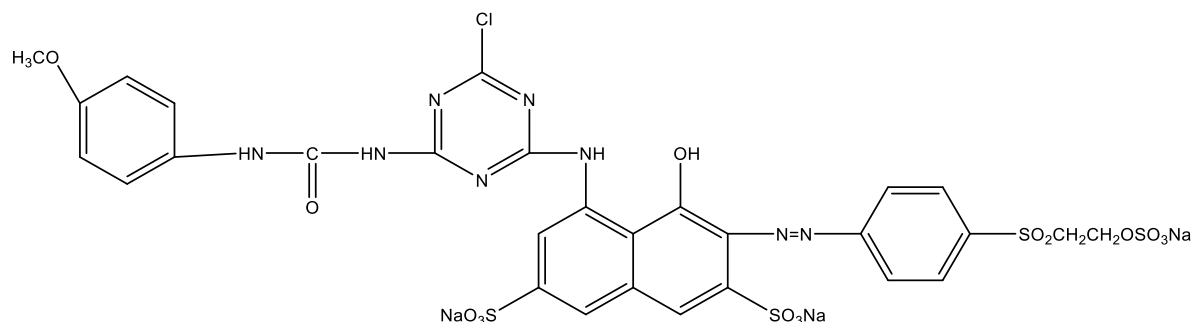
Figure 7: Effect of dyeing time (50-70 min.) on % fixation of four reactive dyes

3.5 Relationship of dye structure and fixation

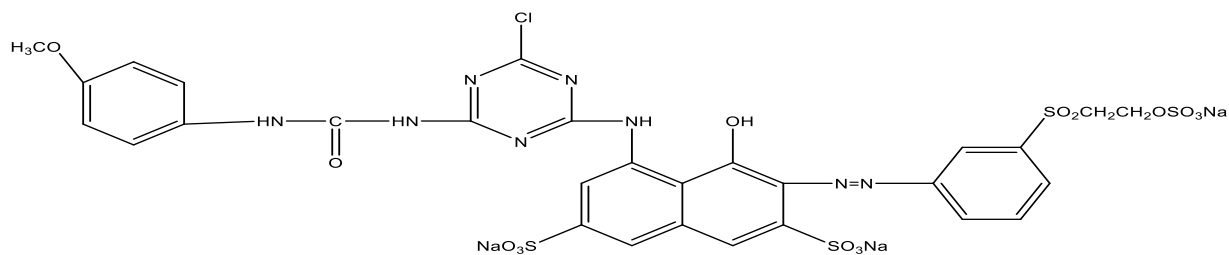
The affinity of the dye for the fiber depends upon the structure of colour molecules in reactive dye. For the cellulosic fibers the hetro-functional active group attending in dye structure have more affinity [34-35]. Dye structure linearity, coplanarity and double bonds are common characteristics in getting maximum dye fixation. In this project, the colour fixation and dye structure relation was studied and results are shown in fig. 9. The dyes D2-D4 having bi-functional reactive groups showed good fixation. The dyes D1 and D3 showed low fixation and dye D2 and dye D4 fixation was higher. It is widely-known that functional group connect with

substrate, and the fixation was heeded for dyes having same functional group with different position showed different colour fixation. Dye 1 and dye 2 have comparable chromophore and linking group but amazing disparate in fixation can be connected with group positions. Para position ester in vinyl sulphone have simple planar structure react with substance causes poor fixation (D1 and D3). The meta position and para position of ester effects on fixation value of dyes due to attachment activity with fiber and hydrolyzed behavior [36]. As yet, dyes having similar functional groups at position put on view different colour fixation. The Linear and planer dye structures of synthesized dyes have considerable affinity and substantivity for cotton. Accordingly, bi-functional azo reactive dyes D2 and D4 exhibited favored exhaust properties than D1 and D3 due to vinyl sulphone reactive group were appeared at meta ester places resulting great planarity and less steric hindrance in the dye structure. Figure 10 presents fiber-dye chemistry. Hydroxyl group of cellulose interface alongside reactive groups of chosen dyes under alkaline condition.

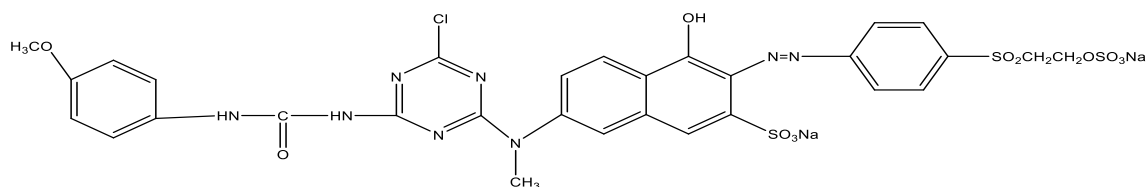
D1



D2



D3



D4

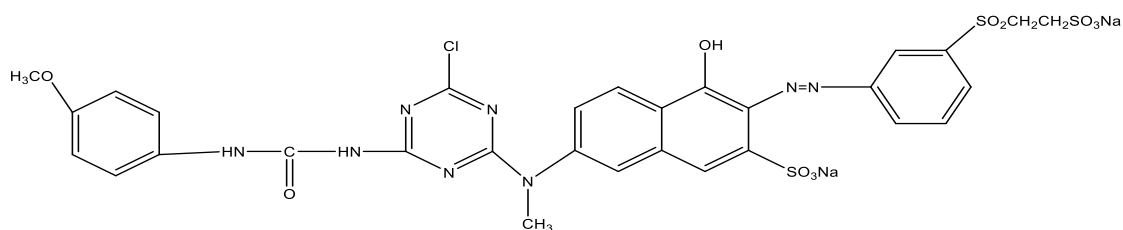


Figure 8: Structure of reactive dyes

Dye no.	Coupling Component	Structural formula
D1-2	H-acid	
D3-4	N-methyl J-acid	

Table 3: Coupling components D1-2 and D3-4, arrow indicates coupling position

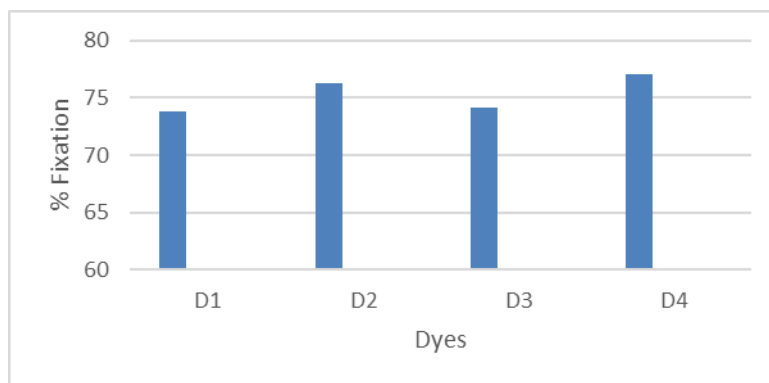


Figure 9: Comparison of the fixation (%) of four reactive dyes

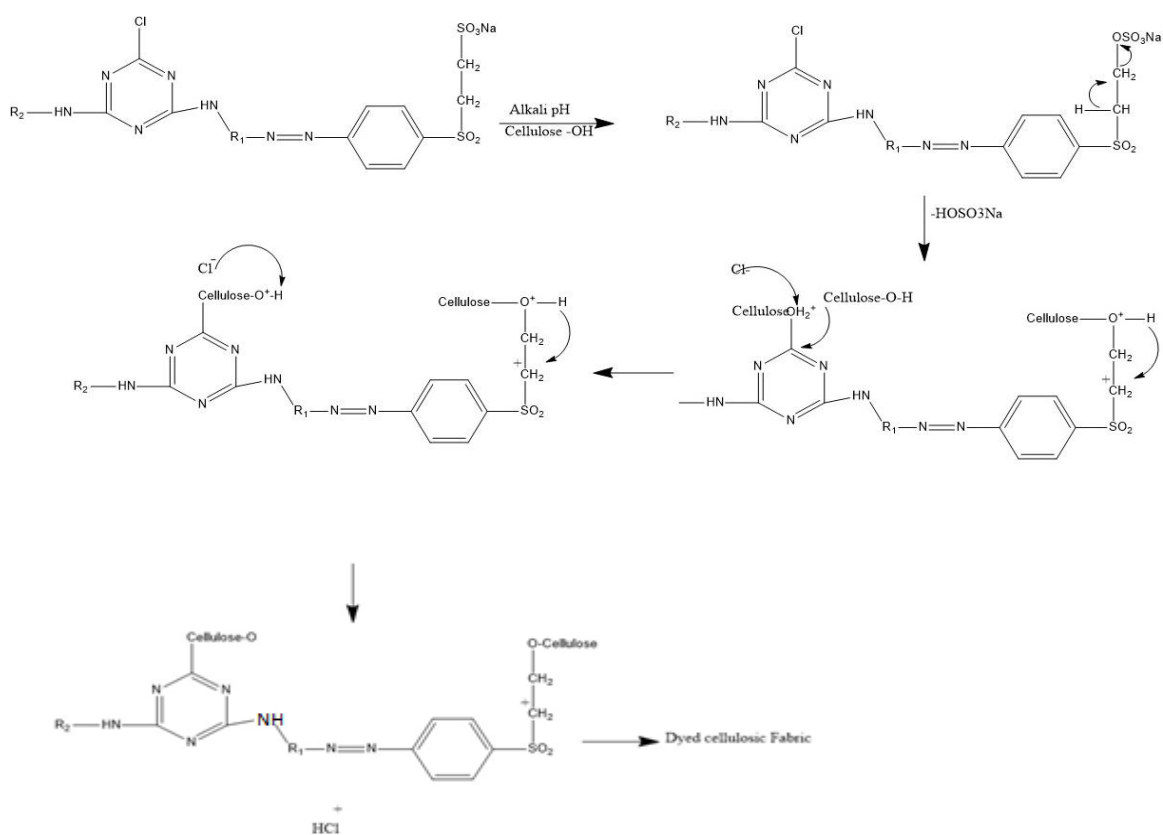


Figure 10: Cotton fibers reaction with synthesized dyes Mechanistic pathway

R1=Coupling Component, R2=1-(4-methoxyphenyl) urea

3.6 Fastness properties

Table 4: Fastness properties of the cotton dyed with four reactive dye





Dye	D-1	D-2	D-3	D-4
Washing fastness	3	4	4	5
Rubbing fastness	4	5	4	5
Light fastness	5	6	5	6

The assessment of fastness properties of four reactive dyes are shown in Table 4. The washing fastness showed that provided good to excellent wash fastness. The fastness properties could be considered to the covalent binding linkages between the dye molecules and the fiber. The chemical fixation of the dye on fabric resulted high wash fastness, D2 and D4 have good fixation with fabric gives excellent washing fastness compare

to D1 and D3. All four dyes rendered good rubbing fastness. D2 and D4 have good penetration and fixation resulted excellent rubbing fastness. The chemical structure of reactive dyes play an important role for light fastness [37]. The chromophore of dye molecules was the great prompting factor and the position of substituents as well considerably the light fastness properties [38]. The azo dyes containing H-acid have great stability of the chromophore because chromophoric group execute to the azo-hydrozone tautomerism that allowed answerable for stability to photo-effect. The stability of reactive dye in under light exposure increased due to strong bond between dye molecules and fiber, the transfer of energy from the excited molecules of dye to the fiber also increased the stability of reactive dye. Vinyl sulphone is stable to acid and CT (Choloro triazine) is stable to alkali which helps to good wet fastness properties [39].

3.7 Colorimetric data of Dyes

Table 5: Colorimetric(CIELab) data of Dyed cotton fabric at 60°C

Dye	Dye Shade	L*	a*	b*	C*	h*	K/S
D1		54.39	55.34	-1.28	55.36	358.67	4.95
D2		53.91	55.13	-1.15	55.15	558.81	5.1
D3		69.39	41.63	61.37	74.15	55.85	7.19
D4		69.06	41.73	61.56	74.37	55.87	7.42

Colorimetric data of Dye 1-4 were compared with each other (Dye 1-2 and Dye 3-4) have same chromophore only change position of ester vinylsulphone at Para and Meta position. The compared result shown in Table 5 the cotton fibers sowed C* value high in D2 compared to D1 indicated dye have excellent dye taken value against Dye 2. K/S value of D2 high against D1 effect on dye strength which was higher side indicated that D2 have more bluer and stronger in shade against D1. The cotton fibers sowed C* value high in D4 compared to D3 indicated dye have excellent dye taken value against Dye 4. K/S value of D4 high against D3 effect on dye strength which was higher side indicated that D4 have more yellower and stronger in shade against D3.

4. Conclusion

Four synthesized reactive dyes have different position of functional group (4-(β -sulfatoethylsulfonyl) aniline) was applied on cotton fabric through Exhaust dyeing. The dyeing parameter like temperature, pH and dyeing time were optimized and dyeing properties were studied. Same dyeing condition all four dyes showed variable exhaustion, fixation and fastness properties. The fixation value and fastness properties of fiber correlated with used functional group (position). D1 and D2 have same chromophore only change in 4-(β -sulfatoethylsulfonyl) aniline used (meta and para), dye D2 have good fixation (78%) and excellent wet fastness compared to D1 (para), same results observed in D4 (meta) have good fixation (73%) and fastness properties compare to D3(para) dyes. Which was assigned to the more planar structure of dyes. In the reactive systems the presents of s-triazine and vinyl sulphone show higher percentage of exhaustion, fixation and fastness properties.

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