



Application of Reactive dyes by Dyeing and Printing Method on Cotton Fabric and Study of Antibacterial Activity

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Abstract

The present work was designed to study bifunctional reactive dyes by printing and cold pad batch method also evaluated their antibacterial activity. The main object of this research paper is to highlight synthesized dyes having excellent fixation abilities for the dyeing and printing industries to further invest upon. All dyes were reported earlier, In the synthesis of reactive dyes phenyl urea derivatives introduced in triazine ring, the dyes have excellent solubility. Synthesized dyes Z1-10 were applied on cotton fabric with cold pad batch dyeing and printing method. The fixation value of dyes in the CPB method and percentage of absorbance in the printing method of dyes were evaluated. Ten synthesized reactive dyes had a 64 % absorbance value in the print method due to the solubility behavior of dyes. The colorimetric data of dyed and printed fabric were examined in the CIELab system. The Fastness properties of dyed and printed fabrics were evaluated through standard test method. Antibacterial activity of synthesized dyes was investigated against different kinds of bacteria on dyed fabric. The synthesized dyes had good Antibacterial affinity compared with selected antibiotics as a reference standard.

Keywords: Bifunctional Reactive dyes, Cotton Fabric, Cold Pad Batch, Printing, Antibacterial activity

1.Introduction

Reactive dyes were largely used for dyeing and printing of cellulosic fibers. Because of the brilliancy of hue and excellent wet fastness properties of reactive dyes most popular for viscose dyeing. Reactive dyes have a chemical bond with the hydroxyl group of cellulose or amino group of protein [1]. Bi-functional reactive dyes are more popular in the textiles industry due to their excellent wet-fastness properties and higher fixation on cotton fibers. Reactive dyes show good wet-fastness because of the presents of a covalent bond with the textile fiber [2]. Reactive dyes were most popular since 1950, a novel structure of reactive dyes and fiber modification was done in textile industries for getting good wet fastness properties as well as fixation value [3]. Several application methodologies were used for dyeing of fabric like exhaust dyeing, cold pad batch and printing method. The exhaust dyeing process is most popular in the dye house. Hydrolysis of reactive dyes cause lots of effluent problems in the textile

industry. It is most important which application method is used for dyeing of cotton fabric [4].

All synthesized dyes were earlier reported. Due to their simplest structure containing a urea group, herein, phenyl urea and its derivatives were directly introduced into triazine ring with the object to obtain reactive dyes with high solubility as well as wet-fastness. In this project, cold pad batch and printing methods were used for the application of synthesized Z1-10 dyes. The present paper deals with the facile and convenient synthesis of phenyl urea derivatives reactive dyes and the study of their dyeing properties, fastness properties and antibacterial activity. Bifunctional reactive dyes have good solubility in the presents of alkali and have good fastness properties [5]. The concentration of alkali salt also affects on fastness properties of reactive dyes [6]. The Cold Pad Batch dyeing process is most suitable for the dyeing of cotton fabric. The cold pad batch method is the most economical method for dyeing cotton fabric with reactive dyes. The energy and water

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consumption are observed minimum in the cpb method and applied without using salts so resulted more ecofriendly method [7]. The cpb method is, very simply for the application of dyeing. The fixation of dyes on cotton fabric generally depends upon the batching time and dwelling time. Microwave irradiation technology is also used for better fixation of dyes in the cpb method [8]. In European countries, mostly cold pad batch method was used now in all Asian countries also accept cold pad batch method [9]. Reactive dyes are water-soluble dyes so the main problem is waste water, this reason it is most important that which method was used for the application of dyes [10]. Reactive dyes have hydrolyzed properties 20 to 30% of dye cannot recycle so it is necessary to use minimum water during application of dyes [11]. Pretreatment of cotton fabric was also important for cold pad batch method, Some pretreatment chemical improves migration properties of dyes on cotton fabric [12]. Sodium alginate thickener is widely used for the application of printing in textile processing industries. For the application of synthesized dyes sodium alginate thickening was used [13].

2. Experimental

2.1 Material and instrument

All chemical products included were obtained from commercial sources. All other chemicals used in synthesis were of chemical grade. Sodium silicate 109⁰ Tw, NaOH 38 Be', sodium alginate, urea, sodium carbonate and resist salt were used for application of dyes. All application work of reactive dyes was done in DyStar Ankleshwar QC laboratory. All chemicals used in the application of cold pad batch and printing provide by DyStar India Pvt Ltd. 100% Cotton scoured, and bleached woven fabric was used for Printing. 100% Cotton scoured and the bleached twill fabric was used for the cold pad batch. Laboratory padding mangle R.B. Electronic model was used for CPB application. Data color 400 TM spectrophotometer was used for color measurement. UV Spectrophotometer SS 5100A Premier Color scan was used to measure maximum absorption (λ_{max}).

2.2 Reactive dye synthesis

Bifunctional reactive dyes Dye Z1-10 has been reported earlier [14]. All synthesized dyes were used for a different type of application on cotton fabrics. The absorbance value of synthesized dyes was measured in water. The maximum absorbance was measured with different solvents i.e. Water, DMF,

Methanol Chloroform, Methanol+ KOH and Methanol + HCL

2.3 Cold pad batch dyeing of synthesized Dyes.

In the application of CPB 20 g L⁻¹ concentration of reactive dyes were used for dyeing of fabric. Horizontal padding mangle was used for the CPB dyeing method. The fabric was passed through the padding mangle where the dye with padding solution. The Batching time of dyed fabric was 16 hrs. In the cold pad batch dyeing method sodium silicate 109⁰ Tw (50 ml L⁻¹), Urea (20 g L⁻¹) and NaOH 38 Be' (0.5 ml L⁻¹) was used as a padding solution. Each sample was instantly wrapped in a plastic bag. The batched fabric was kept in a dark room at room temperature. After batching fabric was first washed with sodium hexa meta phosphate to remove unfixed dyes from dyed fabric, after that cold wash and hot wash is given to the dyed fabric. After removing all unfixed dyes from the fabric, the dyed fabric was kept in the oven in dry at 60⁰C. The dyed fabric was measured on Data Color 400 TM spectrophotometer. The reflectant value L*, a*, b*, C*, H and K/S were recorded on Data Color 400 TM Spectrophotometer. The fixation value of CPB dyed fabric was calculated by below equation.

$$\%F = \frac{\frac{K}{S} \text{ after wash}}{\frac{K}{S} \text{ before wash}} \times 100\% \quad (3)$$

2.4 Printing application of synthesized Dyes.

Sodium alginate thickener was used for the preparation of print paste and a direct style of printing was used. Sodium alginate thickener gives good color strength and wet-fastness properties to print fabric. Reactive Dyes X g KG⁻¹, Thickener 350 g KG⁻¹, Urea 250 g KG⁻¹, sodium carbonate 15 g KG⁻¹, resist salt 6 g KG⁻¹, water 444 g KG⁻¹ total 100 g KG⁻¹ was used for the preparation of print paste [15]. 3% Concentration of dyes were used for printing shade and 1:3 reduction print was used for printing. After the preparation of print paste, the paste was applied on cotton fabric with screen printing. After printing printed fabric dry at 60⁰C in the oven, dried printed fabric fixed on the steamer machine for steaming at 102⁰ C for 10 min.. The printed fabric was washed with nonionic detergent. Printed fabric reflectance measured with Data Color 400 TM Spectrophotometer. Leveling properties of printed fabric measured with the colour differences of each sample at six separate points.

2.5 Measurement of color

Measurement of all dyed and printed fabric was obtained by using a Data Color 400 TM spectrophotometer .D65 illumination,10⁰ standard observer and UV included condition used for measurement. Kubelka-Munk equation used for calculation of K/S values[16]. $K/S=(1-R)^2 / 2R$ where K is absorbance coefficients is scattering coefficient and R is % reflectance. CIELab values of all synthesized dyes were measured. Lightness (L*),Chroma(C*) positive indicate brightness and negative indicate dullness, hue(H*) angle from 0⁰ to 360⁰,a* value show the degree of positive indicate redness and negative indicate greenness and b* perform the degree of positive indicate yellowness and the degree of negative blueness of color.

2.6 Fastness properties

Wash fastness properties of dyed and printed fabric were achieved in consonance with BS 1006 1990 C06 method. the fastness properties of Light were tested according to BS 1006 1990 B02 using Q-SUN Xe-1-Bxenon light fastness machine. Colour changes of all dyed and printed samples were checked visually using Machine Paramount Color Machin. Rubbing fatness of synthesized dyes were tested on Crock Meter (Atlas).

2.7Antibacterial activity

The antibacterial activity of synthesized dyes was examined with used the disc diffusion method on an agar plate. Staphylococcus aureus and Bacillus subtilis bacteria were used as Gram(+) bacteria. Escherichia coli and Pseudomonas aeruginosa were used as Gram(-) bacteria [16], The Bacteria were incubated at 37⁰ C. for 24 hours. The Inhibition Zones of diameter were measured in millimeters standard discs of Ampicillin ,The Ampicillin served as a positive controls and DMSO solvents used as a negative control filter discs impregnated with this solvent. The Mueller-Hinter agar is used as an Agar , Which is rigorously tested for composition and pH. In the plate depth of agar is a factor to be considerable in the disc diffusion method [17].

The antibacterial activity of synthesized dyes and dyed fabric were examined by the disk diffusion method on an agar plate. The blended film samples were cut into 1 cm diameter and brought into 10 ml nutrient agar, in which 10 µl of microbe culture was inoculated. After the Solidification the plates were incubated at 37⁰ C. for 24 hours. After that Inhibition zone of diameter was measured.

3. Results and Discussion

3.1 Synthesis of Dyes (Z1 to Z10)

Bi Functional Reactive dyes Z1-Z10 has been reported earlier [14]. The absorbance value of dyes was observed wavelength of maximum absorption (λ_{max}) was 498 nm to 502 nm. The common structure of synthesized dyes is described as below in figure1 where R was phenyl urea derivatives.

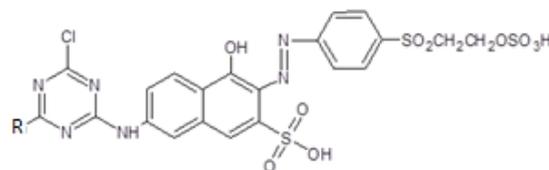


Figure 1. Chemical structure of synthesized reactive dyes

Where R= Phenyl Urea Derivatives

- | | |
|--------------------|----------------------|
| a : Phenyl | f : p-Nitro Phenyl |
| b : o-Tolyl | g : m- Nitro Phenly |
| c : m-Tolyl | h : o-Chloro Phenly |
| d : p-Tolyl | i : m Chloro Phenly |
| e : o-Nitro Phenyl | j : p-Methoxy phenyl |

The absorbance value of synthesized dyes Z1-10 were recorded with used water ,DMF, methanol, DCM, chloroform, methanol+KOH ,methanol+HCL and chloroform+Piperidine. Recorded absorbance data of dyes shown in Table 1 which showed that the band were observed in the region 498-502 nm in water,493-496 nm in DMF,483-486 nm in Methanol,471-475 nm in dichloro methanol and458-461 nm in chloroform. The bathochromic shift was observed in water relative to DMF, methanol, DCM, and Chloroform. The solvent polarity increases due to an increase in the dielectric constant of water corresponding to DMF. Methanol, DCM and chloroform, The results suggest that the excited state in were more polar than the ground state . The effect of acid and base on UV vis spectra is shown in Table 1.In methanol the absorbance spectra of the dyes were quite sensitive to the adding of 0.1 M KOH. By the adding of 0.1 M KOH the λ_{max} of synthesized dyes showed a bathochromic shift, this indicates the dyes were present in different tautomeric form with methanol. With the addition of 0.1 M HCL all dyes showed a large bathochromic shift. This result indicates that the dyes exist in the cationic form in acidic methanol solution. In chloroform and piperidine solution all dyes did not charge significantly.

3.2 Cold Pad-Batch method.

In the CPB method 20 g L⁻¹ concentrations of dyes were used. It was observed that the good fixation and the levelness of dyeing achieved on fabric with synthesized reactive dyes. Figure 2 shows that Dyes Z1-Z10 had a fixation 76.3 to 77.3 %. This dye has good solubility due to the presence of Phenyl Urea group and this reason effect shows in the fixation value of dyes. The synthesized all dyes were Yellow to Orange in the shade. Vinyl Sulphone base yellow reactive dyes have good absorbance and fixation value [18].

The CPB dyeing results for Z1-10 are provided in Table 2. To analogize the effect of the phenyl urea group on the CPB dyeing properties of synthesized dyes. As shown in Table 2, we can see that the fixation of dye Z1 was 77.3%. Using the soluble group in the dye molecule, the solubility of the dye Z1 was found to be naturally higher than that of other dyes. Being to the high solubility of the dye Z1, its fixation was higher than that of the other dyes.

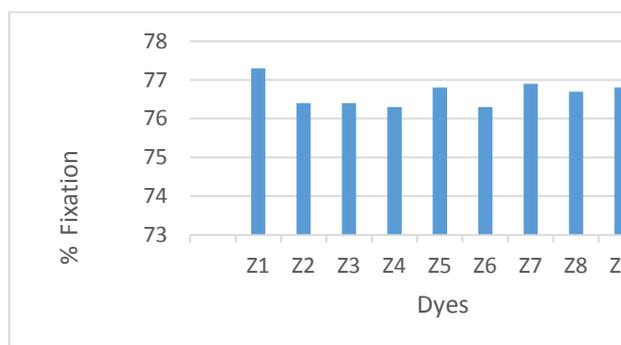


Figure 2. Dye fixation by CPB method

The colorimetric (CIELab) values of the dyed samples are given in Tables 3. The synthesized dyes have a good affinity to cotton fibers with the CPB method. The color strength (K/S) of all dyes for cotton fabric followed the subsequent order.

Z1 > Z2 > Z8 > Z10 > Z7 > Z9 > Z4 > Z3 > Z5 > Z6

Dye Z1 having the highest value of color strength (K/S) and dye Z6 having the lowest value of color strength (K/S).

3.3 Printing Method

Synthesized reactive dyes were applied to a cotton fabric with the printing method. 3% of dye concentration was used to print the fabric. It is clearly shown in Figure 3 that the Absorbance value of dyes was average 64.1% on cotton fabric. The levelness value of printed fabric was achieved for synthesized dyes. K/S Values of print fabric and CIELab Values

of the printed samples are given in Table 3. The fixation value of printed fabrics were examined by equation no 3. Figure 3 showed that the fixation value was higher in Z1 dyes which have containing phenyl urea group in dye molecules, resulted good solubility and high fixation value.

The color of a printing on cotton fibers are shown in terms of CIELab value in Table 4. The data of Dyes Z1-10 summarized in Table 2 showed that the color strength of all dyes followed the following order.

Z1 > Z5 > Z7 > Z2 > Z10 > Z4 > Z6 > Z9 > Z8 > Z3

Dye Z1 having the highest value of colour strength (K/S) and dye Z3 having the lowest value of color strength (K/S).

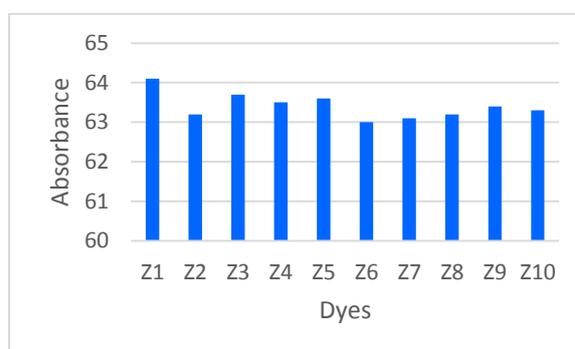


Figure 3. After printing percentage absorbance of dyes

4. Fastness properties of Synthesized dyes

The color fastness of reactive dyes depends on the solubility of dyes and the presence of the covalent bond [19]. The washing fastness of reactive dyes depends upon the chemical fixation of the dye molecules [20]. In Table 5 colour change, light-fastness, wash-fastness and rubbing-fastness of the dyed and printed fabric of dyes Z1-10 is given. Light-fastness of dyed fabric achieved moderate to good. Wash-fastness of both CPB and Print fabric shows good to excellent. Rubbing Fastness of CPB and printing fabric wet as well dry achieved Moderate to Good. Because of the higher water solubility of dyes by reason of the introduction of phenyl urea and its derivatives, the color fastness test displayed that the rub-fastness and wash-fastness of the dye Z1 containing phenyl urea was slightly superior to other dyes. The high water-solubility provided the dyes besides a good wash-off behavior, which was agreeable to upgrade the wet-fastness and rub-fastness. Furthermore, the light-fastness nature of the dyes Z1 containing phenyl urea was 4-5 grade, which was a higher grade than other dyes.

5. Antibacterial Activity

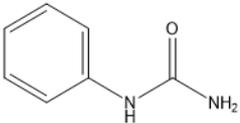
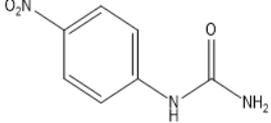
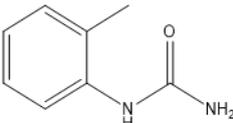
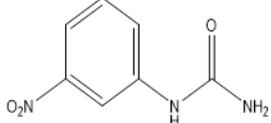
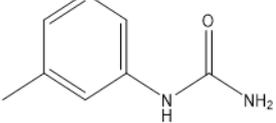
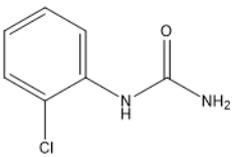
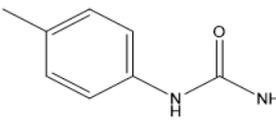
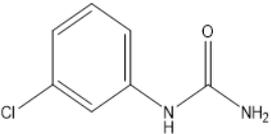
The synthesized dyes were examined using the disc diffusion method as shown in Table No 6. Two stain of bacteria were used. Ampicillin is used as an anti-bacterial. From the antibacterial activity all dyes showing good antibacterial activity are shown in Table. The antibacterial affinity of all Z1-10 was good against two stains of bacteria. Dye Z3 showed

excellent activity against *Escherichia coli* with respect to standard Ampicillin. Dye Z9 showed excellent activity against *Pseudomonas* with respect to standard Ampicillin. Z5 Dye showed very good activity against *Bacillus subtilis* with respect to standard Ampicillin. Dye Z1, Z4, Z8 and Z10 showed excellent activity against *Staphylococcus aureus* with respect to Ampicillin.

Table 1. The visible absorbance spectra of dyes

Dye	Water	DMF	Methanol	DCM	Chloroform	Metanol+KOH	Metanol+HCL	Chloroform+Piperidine
Z1	499	494	484	474	459	499	511	461
Z2	500	495	485	475	460	500	512	462
Z3	498	493	483	473	458	498	511	461
Z4	499	494	485	474	459	499	511	461
Z5	502	496	486	477	461	502	514	464
Z6	498	493	483	473	458	498	511	461
Z7	498	493	483	471	458	498	513	464
Z8	498	493	483	473	458	498	511	461
Z9	501	496	486	476	461	501	513	463
Z10	499	494	486	474	459	499	511	462

Table 2. Fixation value of dye Z1-10

Dye	R	% Fixation	Dye	R	% Fixation
Z1		77.3	Z6		76.3
Z2		76.4	Z7		76.9
Z3		76.4	Z8		76.6
Z4		76.3	Z9		76.8

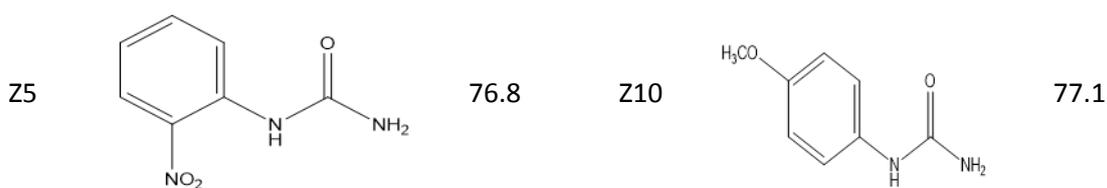


Table 3. Colorimetric (CIELab) data of dyes ZI-10 on cotton by CPB method

Dyes	K/S	L*	a*	b*	C*	H*
Z1	90.01	80.63	13.47	84.17	85.24	80.91
Z2	89.05	80.74	13.3	84.11	85.15	81.02
Z3	86.01	80.71	13.08	83.17	84.19	81.06
Z4	87.85	80.76	13.24	83.38	84.41	81.02
Z5	85.63	80.83	13.13	83.38	84.41	81.05
Z6	85.31	80.79	13.04	83.22	84.24	81.1
Z7	88.35	80.73	13.16	83.84	84.87	81.08
Z8	89.05	80.8	13.23	84.23	85.27	81.07
Z9	87.97	80.64	13.22	83.62	84.66	81.01
Z10	88.65	80.68	13.32	83.79	84.84	80.97

Table 4. Colorimetric (CIELab) data of dyes ZI-10 on cotton by printing method

Dyes	K/S	L*	a*	b*	C*	H*
Z1	88.79	72.61	31	72.37	78.73	66.81
Z2	86.47	72.75	30.85	71.94	78.28	66.79
Z3	85.18	80.66	13.26	82.98	84.04	80.92
Z4	86.31	72.73	30.87	71.87	78.21	66.76
Z5	87.53	72.65	30.95	72.07	78.43	66.78
Z6	86.25	80.64	13.24	83.21	84.26	80.96
Z7	87.08	72.6	30.97	71.83	78.22	66.68
Z8	85.97	72.67	30.85	71.64	78	66.68
Z9	86.01	76.72	21.54	77.96	80.88	74.55
Z10	86.32	76.7	21.63	78	80.94	74.5

Table 5. Fastness results after CPB and printing method

Dye	CC	Light Fastness		Wash Fastness		Rubbing Fastness			
						Wet		Dry	
		CPB	Printing	CPB	Printing	CPB	Printing	CPB	Printing
Z1	5	4	5	5	4	4	4	5	4
Z2	4	4	4	4	4	4	3	3	4
Z3	4	3	4	4	4	3	3	3	4
Z4	4	4	3	4	3	4	4	4	4

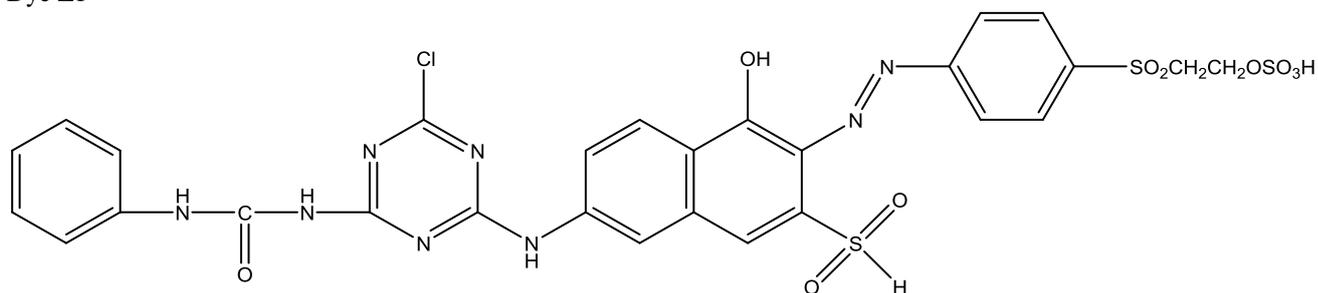
Z5	4	3	4	4	4	3	4	4	4
Z6	4	4	4	4	4	3	4	4	4
Z7	4	3	3	4	3	4	3	4	4
Z8	4	4	3	4	4	4	3	4	4
Z9	4	4	4	4	4	4	4	4	4
Z10	3	4	4	3	4	4	4	5	4

Light fastness : 1-poor: 2-Slight: 3-Morderate: 4 -Fair: 5-Good: 6-Very good: 7-Excellent: 8-maximum

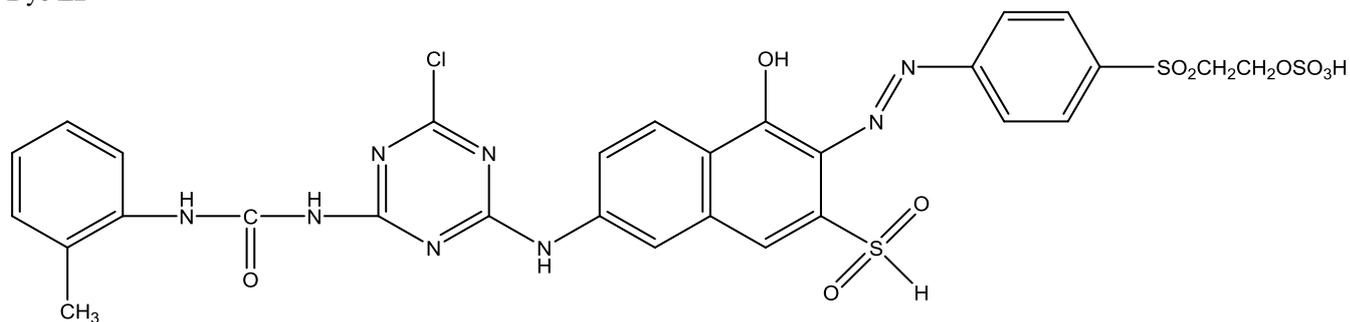
wash and Rubbing fastness : 1-poor: 2-Fair: 3-Good: 4 -Very good: 5-Excellent

CC: colour change

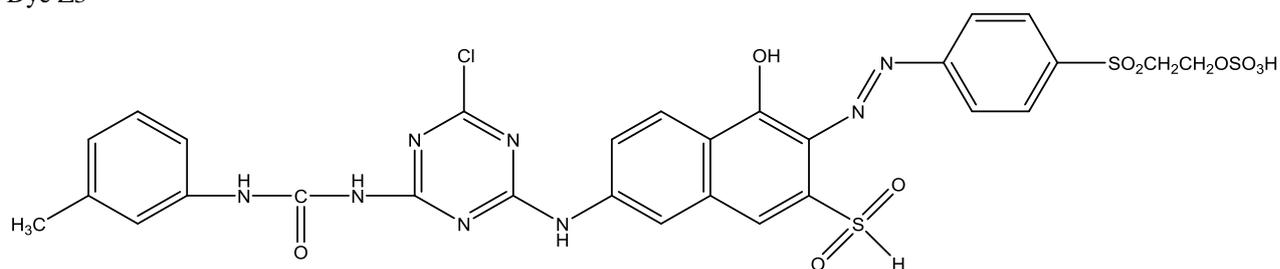
Dye Z1



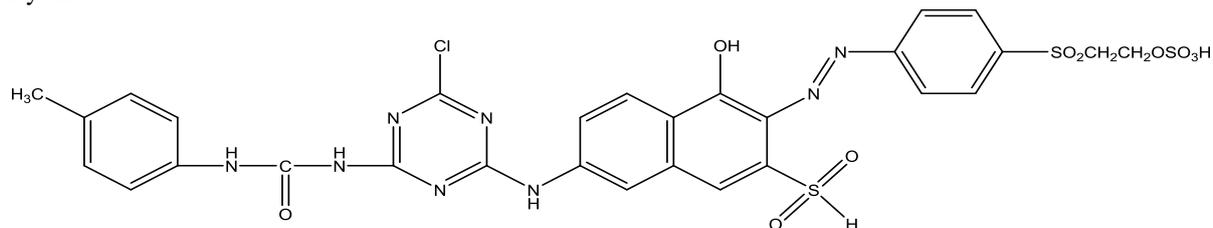
Dye Z2



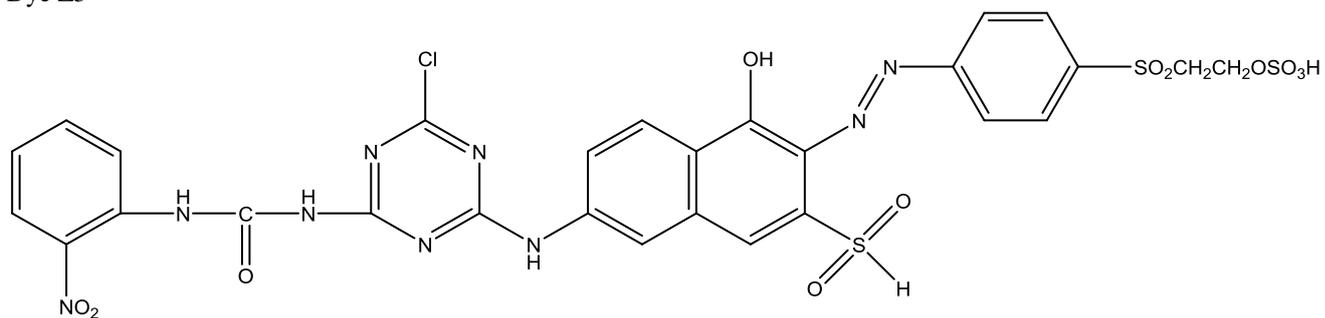
Dye Z3



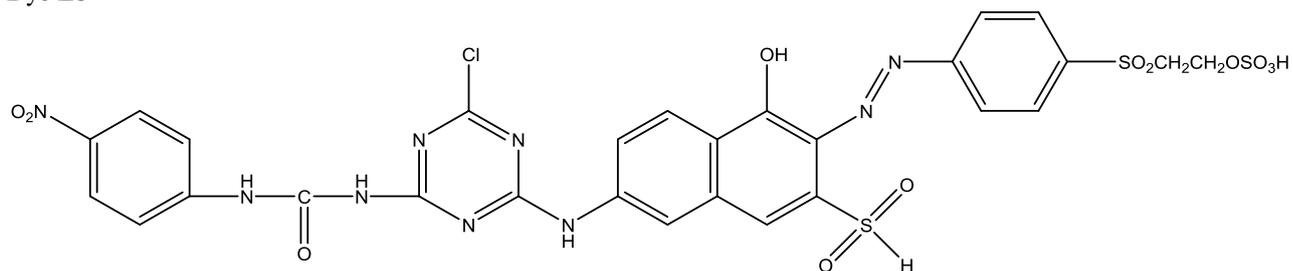
Dye Z4



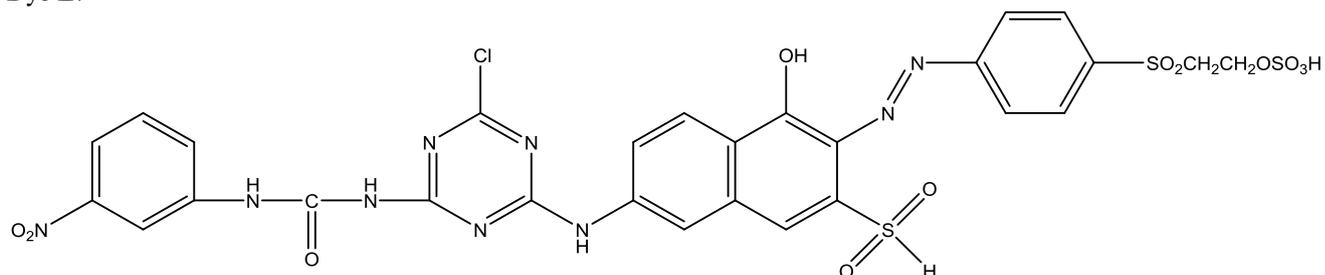
Dye Z5



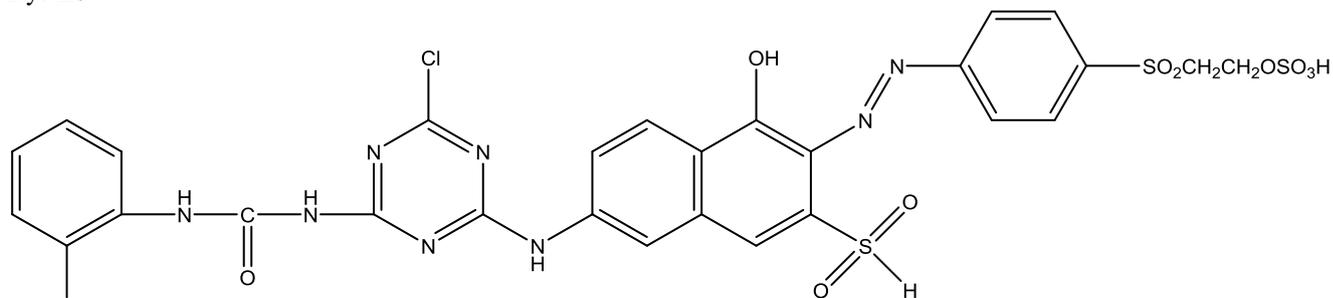
Dye Z6



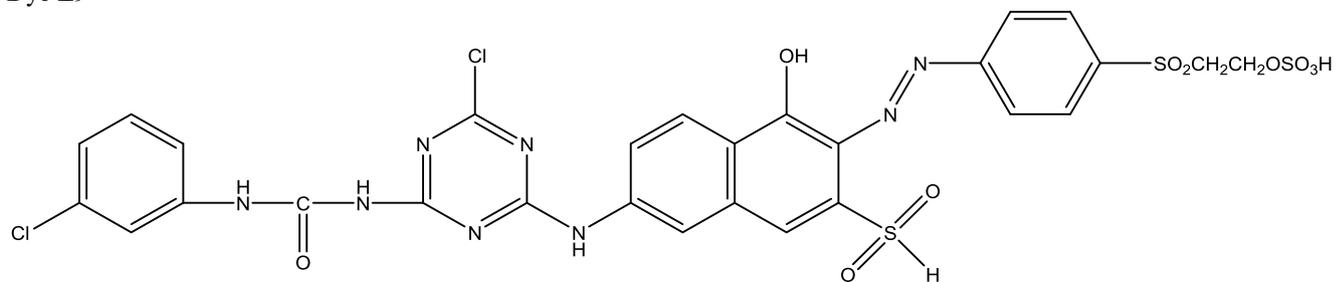
Dye Z7



Dye Z8



Dye Z9



Dye Z10

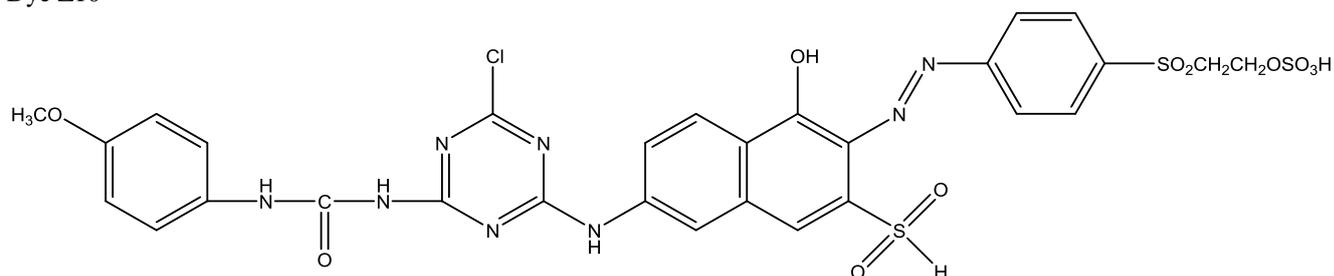


Table 6. Antibacterial activity data of dyes Z1 -10

Sample	Inhibition Zone (mm/mg sample) Concentration 2mg/disc			
	G-		G+	
	Escherichia coli	Pseudomonas aeruginosa	Bacillus subtilis	Staphylococcus aureus
Ampicillin	26	27	27	21
Z1	15	17	16	17
Z2	16	15	17	15
Z3	18	16	15	16
Z4	15	16	17	17
Z5	13	16	18	15
Z6	17	17	15	16
Z7	16	15	17	16
Z8	15	17	15	17
Z9	13	18	16	16
Z10	15	15	16	17

6. Conclusion

A series of bi-functional reactive dyes Z1-10 based on phenyl urea derivatives were earlier reported. All dyes were light orange to yellow in Colour. The cotton fiber was dyed and printed with ten bi-functional reactive dyes (which have different phenyl urea derivatives). The application of dyes was examined by cold pad batch and printing method on cotton fabric. The dyes have a good Fixation value in cpb (76-77%) and printing (64%) method. The fixation of the dyes increased by the introduction of phenyl urea and their derivatives: however the fixation value of dye Z1 was better than phenyl urea

derivatives. According to the fastness test, dyes substituted with phenyl urea displayed superior wash-fastness than the other Z2-10 dyes. The fastness properties of all dyes were evaluated in both CPB and Printing method. Antibacterial activities of synthesized dyes were evaluated. All dyes showed excellent activities against using bacteria strain with respect to Ampicillin.

Conflicts of interest

“There are no conflicts to declare”.

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