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## Research Article

### SYNTHESIS OF SOME MONO AZO REACTIVE DYES HAVING QUINAZOLIN-4(3H)-ONE MOIETY AND THEIR APPLICATIONS ON VARIOUS FIBRES

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#### ABSTRACT

Some new mono azo dye derivatives having Quinazolin-4(3H)-one, 4,4'-(1,3,4-Oxadiazole-2,5-diyl) dianiline and 2,4,6-Trichloro-1,3,5-triazine moieties were synthesized and were evaluated as reactive dyes for the exhaust dyeing of silk, wool and cotton fiber. The title dyes were synthesized from reactions of diazonium salts of 3-(4-(5-(4-Aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one with various cyanurated coupling components. The synthesized dyes were applied to silk, wool and cotton fiber and their color fastness to light, washing, and rubbing were also measured. The dyes showed good to moderate affinity to cotton, wool and silk fiber. Spectral properties and colorimetric data ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $c^*$ ,  $H^*$  and  $K/S$ ) have also been investigated.

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#### INTRODUCTION

Reactive dyes are coloured compounds which contain one or two groups capable of forming covalent bonds between a carbon or phosphorus atom of the dye ion or molecule and an oxygen, nitrogen or sulphur atom of a hydroxy, an amino or a mercapto group, respectively, of the substrate. Such covalent bonds are formed with the amino, hydroxyl groups of cellulosic fibres, with the amino, hydroxyl and mercapto groups of protein fibres and with the amino groups of polyamides (Zollinger, 1991). Reactive dyes are extensively used in the textile industry not only for cotton, but also for wool and silk because of their wide variety of color shades, high wetfastness, ease of application and brilliant colors. A steady increase in the reactive dye usage has been observed as a result of the increased cotton use worldwide (Epolito et al., 2005).

Quinazolinones are very important class of heterocyclic carrier systems of reactive dyes due to their high chromophoric potential. As a part of our continuous interest in the reactivity of Quinazolinone derivatives we have synthesized some novel reactive dyes having quinazolin-4(3H)-one moiety and investigated their applications as reactive dyes to silk, wool and cotton fiber (Patel et al., 2010; Patel et al., 2010; Patel et al.,

2009). Here we report the synthesis of some new mono azo dyes and their dyeing properties.

#### MATERIALS AND METHODS

Commercially available silk, wool and cotton fibers were used for dyeing. The chemicals and reagents were obtained from Sigma-Aldrich and all the coupling components were received from Atul Ltd., Valsad, Gujarat, India and used without further purification.

All melting points taken by open capillary method are uncorrected and are expressed in °C. TLC analysis was carried out on silica gel G F254-precoated aluminum sheets (Fried and Sherma, 1982). IR spectra were recorded on a Shimadzu Model 8400S system using the potassium bromide wafer technique. <sup>1</sup>H-NMR (400 MHz) and spectra were determined on Bruker Avance II in DMSO solvent using TMS as internal standard, UV-vis absorption spectra were recorded on a Thermo Scientific Evolution 300 Spectrophotometer at the wavelength of maximum absorption ( $\lambda_{max}$ ) using water as solvent. Elemental analysis (C, H, and N) were carried out using a CHN analyser, Carlo Erba, Italy. The dyeing was done by using a Laboratory Rota Dyer instrument. Colorimetric data ( $L^*$ ,  $a^*$ ,

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b\*, C\*, H\* and K/S) were recorded on a Reflectance Spectrophotometer Gretag Macbeth CE: 7000.

### **Synthesis of Reactive Dyes**

#### **Step 1: Synthesis of 2-Phenyl-4H-benzo[d][1,3]oxazin-4-one [Patel et al., 2010]**

To a stirred solution of 2-Aminobenzoic acid (1.37 g, 0.01 mol) in pyridine (60 ml), Benzoyl chloride (1.16 ml, 0.01 mol) was added dropwise, maintaining the temperature near 0-5 °C or 1 hour. The reaction mixture was stirred for another 2 hours at room temperature until a solid product was formed. The reaction mixture was neutralized with saturated Na<sub>2</sub>CO<sub>3</sub> solution. A yellow separated solid was filtered, washed with water, and recrystallized from ethanol. Yield: 86 %, m. p. 192-193 °C.

#### **Step 2: Synthesis of 4,4'-(1,3,4-Oxadiazole-2,5-diyl)dianiline [Patel et al., 2010]**

4-Amino benzoic acid (1.37 g, 0.01 mol) and 50 ml methanol was refluxed on water bath for 3 hours with a few drops of concentrated H<sub>2</sub>SO<sub>4</sub> as a catalyst. After completion of the reaction, reaction mixture was poured onto ice cold water. The obtained solid i.e. Methyl 4-aminobenzoate was washed with NaHCO<sub>3</sub> solution (5%), dried and recrystallized from methanol. Methyl 4-aminobenzoate (1.51 g, 0.01 mol) in 30 ml ethanol was added to hydrazine hydrate (0.015 mol). The reaction mixture was refluxed on a water bath for 4 hours. After completion of reaction, excess ethanol was distilled off and the reaction mass was poured onto ice cold water. The obtained solid i.e. 4-Aminobenzohydrazide was filtered, washed and recrystallized from methanol. 4-Aminobenzohydrazide (1.51 g, 0.01 mol) and 4-amino benzoic acid (1.37 g, 0.01 mol) was refluxed in 5 ml POCl<sub>3</sub> for 8 hours. After completion of the reaction, reaction mixture was poured onto ice cold water. The obtained solid i.e. 4,4'-(1,3,4-Oxadiazole-2,5-diyl) dianiline was washed with NaHCO<sub>3</sub> solution (5%), dried and recrystallized from methanol. Yield: 75 %, m. p. 428-430 °C.

#### **Step 3: Synthesis of 3-(4-(5-(4-Aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one [Gao et al., 2007]**

2-Phenyl-4H-benzo[d][1,3]oxazin-4-one (2.23 g, 0.01 mol) and 4,4'-(1,3,4-Oxadiazole-2,5-diyl)dianiline (2.52 g, 0.01 mol) were dissolved in pyridine (40 ml) and heated under reflux for 6 hours under anhydrous reaction conditions and then allowed to cool at room temperature. The reaction mixture was then treated with ice cooled dilute HCl solution and stirred. A solid separated out which was filtered off and washed with water to remove any adhered pyridine. The crude Quinazolinone thus obtained was dried under vacuum and recrystallized from ethanol.

Yield: 88 %, m. p. above 450 °C.

#### **Step 4: Diazotization of 3-(4-(5-(4-Aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one [Mistry et al., 2011]**

Sodium nitrite (0.69 g, 0.01mol) was added to a cooled concentrated sulphuric acid (5 ml) and then the mixture was warmed gradually on water bath at 65-70 °C. The solution was

cooled to 0-5 °C and then a mixture of propionic acid (2 ml) and acetic acid (3 ml) was added to it. 3-(4-(5-(4-aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one (4.57 g, 0.01 mol) was added gradually to a nitrosyl sulphuric acid paste at 0 °C, and then stirred for 30 minutes, maintaining the temperature at 0-5 °C. After completion of reaction, excess of nitrous acid was destroyed using sulphamic acid.

The diazotized solution at 0-5 °C was obtained and used for subsequent coupling reaction.

#### **Step 5: Synthesis of 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphato Cyanurated H-acid Cyanuration of H-acid [Patel et al., 2010]**

Cyanuric chloride (1.85 g, 0.01 mol) was stirred in acetone (25 ml) at a temperature below 5 °C for a period of an hour. A neutral solution of H-acid (3.19 g, 0.01 mol) in aqueous sodium carbonate solution (10% w/v) was then added in small lots in about an hour. The pH was maintained neutral by simultaneous addition of sodium carbonate solution (1% w/v). The reaction mass was stirred at 0-5 °C for further 4 hours. The Cyanurated H-acid solution was used for subsequent coupling reaction.

#### **Condensation with 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphate**

The temperature of ice-cooled well stirred solution of Cyanurated H-acid was gradually raised to 45 °C. 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphate (2.81 g, 0.01 mol) was added slowly to the Cyanurated H-acid solution at same temperature during a period of 30 minutes, maintaining the pH neutral by simultaneous addition of sodium carbonate solution (1% w/v). After the addition was completed, stirring was continued for further 3 hours. The 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphato Cyanurated H-acid solution thus obtained was subsequently used for further coupling reaction.

#### **Step 6: Coupling of diazotized solution with 2-((4-Aminophenyl)sulphonyl)ethyl hydrogen sulphato Cyanurated H-acid coupling component: Synthesis of dyes (D<sub>1</sub> to D<sub>12</sub>)**

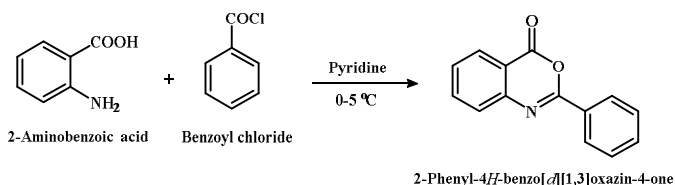
The above mentioned freshly prepared diazotized solution was added to an ice cooled and well stirred solution of 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphato Cyanurated H-acid over a period of 10-15 minutes. The pH was maintained at 8.0 to 8.5 by simultaneous addition of sodium carbonate solution (10% w/v) and stirring was continued for 4 hours. Sodium chloride (15 g) was then added and the mixture was stirred for an hour. The solid dye separated out was filtered, washed with minimum amount of acetone and dried.

Following the above procedure, other reactive dyes SF<sub>2</sub> to SF<sub>12</sub> were synthesized by using various Cyanurated coupling components such as J-acid, N-Methyl J-acid, Gamma acid, Peri acid, Sulpho Tobias acid, Koch acid, Bronner's acid, Chicago acid, K-acid, N-Phenyl J-acid and M-acid.

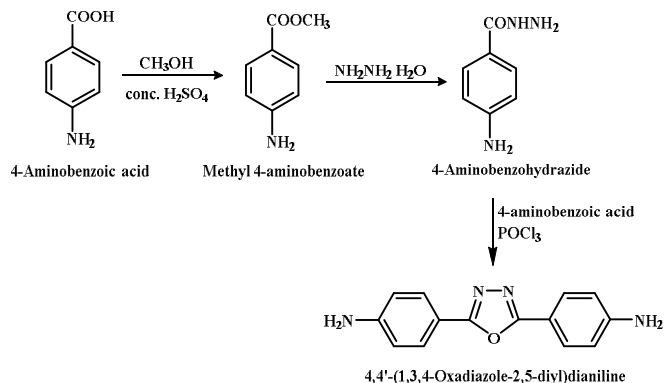
All the synthesized dyes were recorded in Table1.

### **Reaction Scheme**

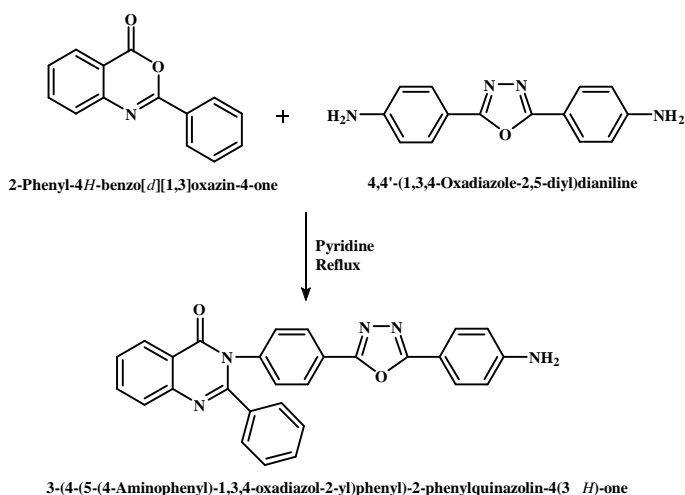
#### **Step 1 Synthesis of 2-Phenyl-4H-benzo[d][1,3]oxazin-4-one**



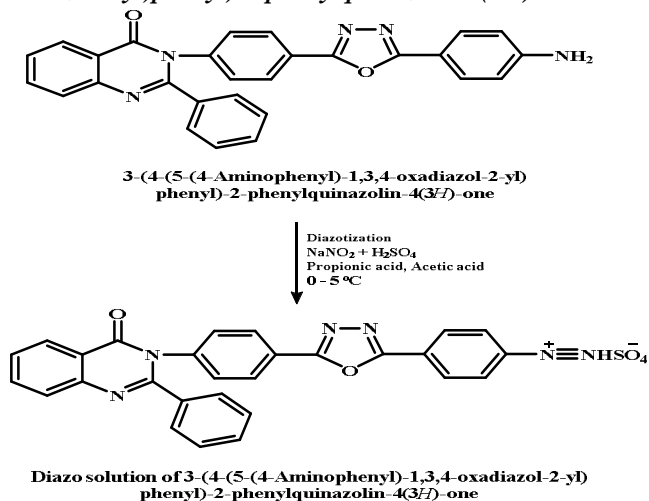
**Step 2 Synthesis of 4,4'-(1,3,4-Oxadiazole-2,5-diyl)dianiline**



**Step 3: Synthesis of 3-(4-(5-(4-Aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one**

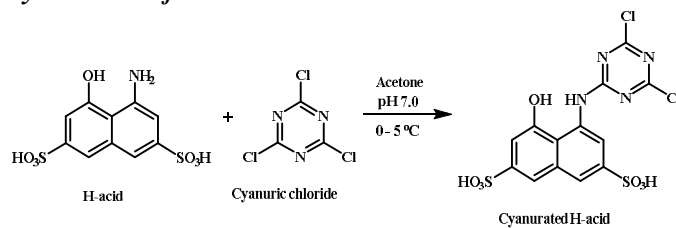


**Step 4: Diazotization of 3-(4-(5-(4-Aminophenyl)-1,3,4-oxadiazol-2-yl)phenyl)-2-phenylquinazolin-4(3H)-one**

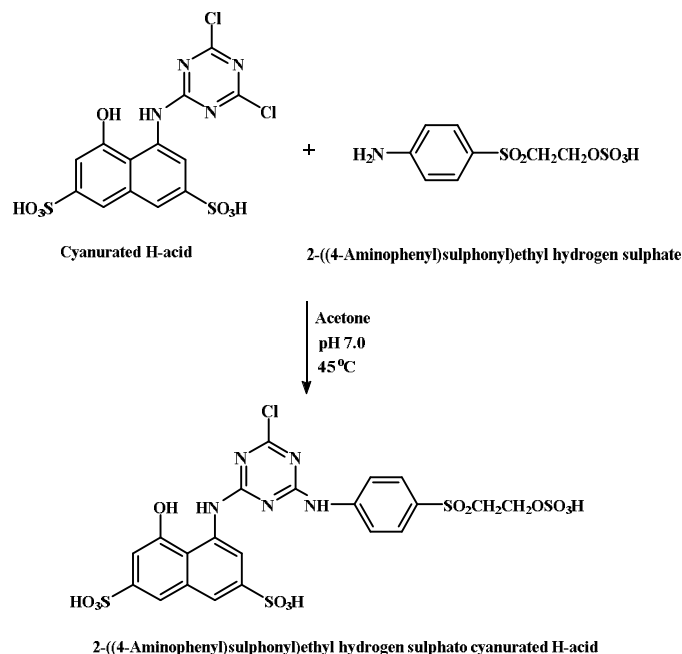


**Step 5: Synthesis of 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphato Cyanurated H-acid**

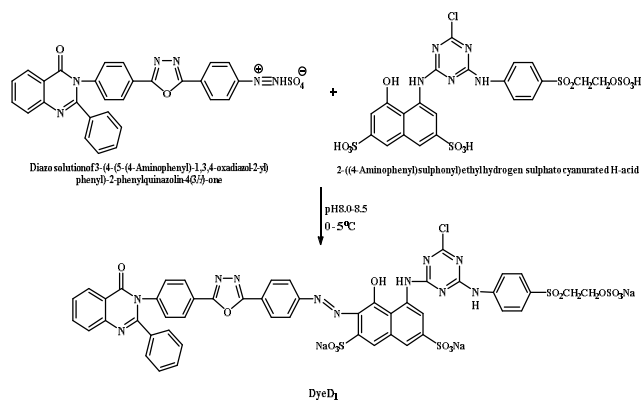
**Cyanuration of H-acid**



**Condensation with 2-((4-aminophenyl)sulphonyl)ethyl hydrogen sulphate**



**Step 6: Coupling of diazotized solution with 2-((4-Aminophenyl)sulphonyl)ethyl hydrogensulphato Cyanurated H-acid coupling component: Synthesis of dyes (D<sub>1</sub> to D<sub>12</sub>)**



**RESULT AND DISCUSSION**

**IR, <sup>1</sup>H NMR Spectral Data**

The structures of dyes were confirmed by various spectroscopic techniques, including IR (Colthup, 1991) and <sup>1</sup>H-NMR (Bassler, 1991) spectral data.

IR spectra of the dyes showed medium characteristic band at 3240-3485 cm<sup>-1</sup> region corresponding to the stretching vibration of -NH- group. Dyes D<sub>1</sub>-D<sub>4</sub> and D<sub>10</sub>-D<sub>12</sub> showed

broad band at 3372-3427  $\text{cm}^{-1}$  region corresponding to the stretching vibration of -OH group.

electron donating or electron attracting group in the dye molecule results in additional color shifts.

Dye No.	Coupling component (R)	Molecular Formula	Mol. Weight g/mol	Yield (%)	m. p. °C	Nitrogen %		Rf Value
						Found	Req.	
D <sub>1</sub>	H-acid	C <sub>49</sub> H <sub>31</sub> ClN <sub>11</sub> Na <sub>3</sub> O <sub>15</sub> S <sub>4</sub>	1246.51	81	>300	12.30	12.36	0.56
D <sub>2</sub>	J-acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>12</sub> S <sub>3</sub>	1144.47	78	>300	13.39	13.46	0.60
D <sub>3</sub>	N-Methyl J-acid	C <sub>50</sub> H <sub>34</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>12</sub> S <sub>3</sub>	1158.50	84	>300	13.22	13.30	0.58
D <sub>4</sub>	Gamma acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>12</sub> S <sub>3</sub>	1144.47	76	>300	13.40	13.46	0.62
D <sub>5</sub>	Peri acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>11</sub> S <sub>3</sub>	1128.47	86	>300	13.56	13.65	0.54
D <sub>6</sub>	Sulpho Tobias acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>11</sub> S <sub>3</sub>	1128.47	78	>300	13.57	13.65	0.52
D <sub>7</sub>	Koch acid	C <sub>49</sub> H <sub>30</sub> ClN <sub>11</sub> Na <sub>4</sub> O <sub>17</sub> S <sub>5</sub>	1332.55	82	>300	11.50	11.56	0.60
D <sub>8</sub>	Bronner's acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>11</sub> S <sub>3</sub>	1128.47	77	>300	13.59	13.65	0.57
D <sub>9</sub>	Chicago acid	C <sub>49</sub> H <sub>31</sub> ClN <sub>11</sub> Na <sub>3</sub> O <sub>15</sub> S <sub>4</sub>	1246.51	81	>300	12.28	12.36	0.62
D <sub>10</sub>	K-acid	C <sub>49</sub> H <sub>31</sub> ClN <sub>11</sub> Na <sub>3</sub> O <sub>15</sub> S <sub>4</sub>	1246.51	79	>300	12.30	12.36	0.58
D <sub>11</sub>	N-Phenyl J-acid	C <sub>55</sub> H <sub>36</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>12</sub> S <sub>3</sub>	1220.57	86	>300	12.54	12.62	0.56
D <sub>12</sub>	M-acid	C <sub>49</sub> H <sub>32</sub> ClN <sub>11</sub> Na <sub>2</sub> O <sub>12</sub> S <sub>3</sub>	1144.47	79	>300	13.40	13.46	0.54

A sharp band observed at 1710- 1720  $\text{cm}^{-1}$  region was due to the stretching vibration of quinazoline ring (amide). Dyes D<sub>1</sub>-D<sub>12</sub> showed carbonyl stretching vibration of quinazoline ring ( $\delta$ -lactum) at 1665-1686  $\text{cm}^{-1}$ , which confirm the conversion of benzoxazine ring to quinazoline ring. The quinazoline ring is further confirmed by the band at 1597-1605  $\text{cm}^{-1}$  showing the stretching vibration of C=N ring of quinazoline moiety. The 1,3,4-oxadiazole ring is confirmed by the band at 1280-1295  $\text{cm}^{-1}$  of C-O-C stretching vibrations. Other bands at 1385-1412  $\text{cm}^{-1}$  and 1156-1195  $\text{cm}^{-1}$  showed the asymmetric and symmetric stretching vibration of sulfonic acid group. IR spectral data of some dyes are given in Table II.

The <sup>1</sup>H-NMR spectra of dyes D<sub>1</sub>-D<sub>4</sub> and D<sub>9</sub>-D<sub>12</sub> showed singlet at 9.0-9.65  $\delta$  ppm was due to the phenolic proton. Dye D<sub>3</sub> showed one more singlet at 3.44  $\delta$  ppm due to the N-methyl proton of coupler moiety. Moreover, dyes D<sub>1</sub>-D<sub>12</sub> showed singlet at 9.43  $\delta$  ppm, which was attributed to NH proton. The aromatic protons (multiplet) were observed from 6.80-8.17  $\delta$  ppm. <sup>1</sup>H-NMR spectral data of some dyes are given in Table III.

Table II IR spectral data of reactive dyes

Dye No.	IR (KBr, $\text{cm}^{-1}$ )
D <sub>1</sub>	3412 (O-H), 3248 (N-H), 3102 (C-H), 1673 (C=O of quinazolinone), 1282 (C-O-C), 1602 (C=N), 1633 (N=N), 1411, 1154 (S=O), 756 (C-Cl),
D <sub>3</sub>	3423 (O-H), 3260 (N-H), 3017 (C-H), 1668 (C=O of quinazolinone), 1283 (C-O-C), 1594 (C=N), 1624 (N=N), 1389, 1175 (S=O), 776 (C-Cl)
D <sub>10</sub>	3419 (O-H), 3244 (N-H), 3081 (C-H), 1673 (C=O of quinazolinone), 1283 (C-O-C), 1607 (C=N), 1624 (N=N), 1387, 1160 (S=O), 775 (C-Cl)

Table III <sup>1</sup>H-NMR spectral data of reactive dyes

Dye No.	<sup>1</sup> H-NMR Chemical shift (DMSO) $\delta$ ppm
D <sub>1</sub>	9.01 (1H, s, -OH), 9.43 (2H, s, -NH-), 3.60-4.09 (4 H, t, -CH <sub>2</sub> ), 6.81-7.87 (24H, m, Ar-H)
D <sub>3</sub>	9.01 (1 H, s, -OH), 9.43 (1 H, s, -NH-), 3.60-4.09 (4 H, t, -CH <sub>2</sub> ), 7.31-7.87 (25 H, m, Ar-H), 3.44 (3 H, s, -CH <sub>3</sub> )
D <sub>10</sub>	9.01 (1 H, s, -OH), 9.43 (2 H, s, -NH-), 3.60-4.09 (4 H, t, -CH <sub>2</sub> ), 6.78-8.17 (24 H, m, Ar-H)

Abbreviation: (s) singlet, (d) doublet, (t) triplet and (m) multiplet.

### Visible Absorption Spectra

The absorption spectra of new dyes D<sub>1</sub>-D<sub>12</sub> were recorded in water at  $4 \times 10^{-6}$  M concentration. The absorption maxima of all the dyes D<sub>1</sub>-D<sub>12</sub> ranged from 410-525 nm. The introduction of

Thus the introduction of methyl group in the dye D<sub>3</sub> resulted in bathochromic shift, same effect is produced by the introduction of phenyl group in dye D<sub>11</sub> which shows bathochromic shift. The visible absorption spectral data of dyes D<sub>1</sub>-D<sub>12</sub> are given in Table IV.

Table IV Physical appearance and absorption maxima of reactive dyes (D<sub>1</sub>-D<sub>12</sub>)

Dye No.	Physical appearance	$\lambda_{\text{max}}$ (nm)
D <sub>1</sub>	Plum purple	525
D <sub>2</sub>	Bronze orange	490
D <sub>3</sub>	Ginger orange	495
D <sub>4</sub>	Brown	410
D <sub>5</sub>	Moss green	410
D <sub>6</sub>	Mustard yellow	410
D <sub>7</sub>	Honey yellow	410
D <sub>8</sub>	Medallion yellow	410
D <sub>9</sub>	Yam orange	410
D <sub>10</sub>	Hazelnut tan	410
D <sub>11</sub>	Brown	410
D <sub>12</sub>	Biscotti tan	410

### Application

#### Dyeing and Fastness Determinations

#### Dyeing procedure

The applications and fastness properties of the synthesized dyes have been performed at Atul Limited, Valsad, Gujarat, India. The synthesized dyes were applied to silk, wool and cotton fiber.

#### Dyeing of Silk

The dye (0.2 g) was pasted with a drop of cold water and then about 80 ml of cold water was added and stirred well to give a clear solution. The resulting dye solution was made up to 100 ml with the dye solution (20 ml), acetic acid (2 ml of 10 % v/v) and water 18 ml. The dye bath temperature was maintained at 30 °C and silk fabric (2 g) was entered, and the temperature was raised to 80 °C over 20 min. At this temperature formic acid (1.5 ml of 40 % v/v) was added to the dye bath to achieve good exhaustion. The dyeing was continued for 40 min more and then the dyed material was washed with cold water, soaping and dried.

#### Dyeing of Wool

Dye (0.2 g) was pasted with a few drops of cold water, then about 80 ml cold water was added, and the mixture was stirred

and made up to 100 ml with dye solution (20 ml), acetic acid (1.5 ml of 10% v/v), Glaubers's salt solution (4 ml of 10 % w/v) and water (14.4 ml). A wool fabric (2 g) was introduced in to the dyebath at 30 °C and the temperature was raised up to 80 °C over 20 min. Sulfuric acid (0.4 ml of 10 % v/v) was then added and the dyeing was continued for 40 min more at the same temperature. The material was then removed, rinsed with cold water, soaping and dried.

**Dyeing of Cotton**

Dye (0.2 g) was pasted with a few drops of cold water, then about 80 ml cold water was added, and the mixture was stirred and made up to 100 ml with dye solution (20 ml), Glaubers's salt solution (4 ml of 10% w/v) and water (14.4 ml). A cotton fabric (2 g) was introduced in to the dyebath at 30 °C and the temperature was raised up to 80 °C over 20 min. Soda ash (Na2CO3) solution (0.4 ml of 10 % v/v) was then added to bring about fixation and the dyeing was continued for 40 min more at the same temperature. The material was then removed, rinsed with cold water, soaping and dried.

**Wash-off Process**

The dyed fabrics (silk, wool and cotton) were rinsed in warm water, scoured with 2 g/l Lissapol detergent at 90 °C for 5 min and rinsed again in warm water. The dyed fabrics afforded color in the first warm water rinse, less color in the scouring bath and practically no color in the second water rinse. This indicated that the unfixed dyes were easily removed from the fiber surface. The amount of the hydrolyzed dye having low substantivity released easily from the substrate after two or three washes. Also higher molecular size greatly increases the wash fastness.

**Fastness determination**

Color fastness to light, washing, perspiration, and rubbing of the prepared dyes on the cotton fabric were studied using the standard methods for the assessment color fastness of textile (Anon, 1990). The obtained results were collected in Table V.

previously heated at 50 °C. The specimen was rinsed twice in cold water for 10 min, squeezed, and the composite specimen was opened out and dried in air. The colour alteration of the uncovered portion of the specimen and the staining for both undyed fabrics was assessed using the international Grey scale.

**Fastness to rubbing**

Test assessment was made according to the Grey scale using Crockmeter of atlas electronic type. Dyed fabrics to be tested were placed on the base of the Crockmeter. A square of white testing cloth was mounting over the end of the finger which protects downward on the specimen sliding back, and force to make ten complete turns of the crank at the rate of one turn per a second. For wet rubbing test, the testing squares were thoroughly wet in distilled water and squeezed between filter papers through hand wringer under standard conditions.

**Fastness to light**

The specimen of the dyed textiles were exposed, in a well ventilated exposure chamber to light from a xenon arc, along with dyed wool standards. The air temperature in the chamber was maintained at 30 °C. The effective humidity was maintained at 45 ± 5%. The variation of light intensity over the area covered by specimen and standards should not exceed 20%. The samples and standards were exposed simultaneously under the same conditions for the same time. The samples were viewed in the light from a day-light fluorescent lamp and given a degree in comparison with the relative to Blue scale (1–8) standards of A.A.T.C.C.

**Colorimetric Data (CIE lab Data)**

The color of a dyeing on silk, wool and cotton fibers are expressed in terms of CIE lab values (Table VI) and the following CIE lab coordinates were measured, lightness (L\*), chroma (C\*), hue angle form 0 ° to 360 ° (H), a\* value represent the degree of redness (positive) and greenness (negative) and b\* represents the degree of yellowness (positive) and blueness (negative).

**Table V** Fastness properties data of reactive dyes D<sub>1</sub>-D<sub>12</sub>

Dye No.	Light fastness			Wash fastness			Rubbing fastness					
							Dry			Wet		
	S	W	C	S	W	C	S	W	C	S	W	C
D <sub>1</sub>	5-6	5-6	6	4-5	4-5	4-5	4-5	4	4-5	4	4-5	4-5
D <sub>2</sub>	5-6	5	5-6	4-5	4	4-5	4-5	5	4-5	4	3-4	4-5
D <sub>3</sub>	5	5	5	4	5	4	4-5	4	4	4	3-4	4
D <sub>4</sub>	5	5	5-6	4	4	4-5	4	4	4-5	3-4	4	3-4
D <sub>5</sub>	5	5	5	4	3	4	4	4-5	4	3-4	4	3
D <sub>6</sub>	5	4-5	4-5	3-4	4	4	4	4	4	3	4-5	3
D <sub>7</sub>	4	4-5	4-5	3	4	4-5	3-4	3-4	4	3-4	3	3
D <sub>8</sub>	4-5	5	4	4	4	4-5	4	4-5	3	3-4	4-5	3-4
D <sub>9</sub>	4	5	4	3-4	4	4-5	3	4	3	3-4	4	4
D <sub>10</sub>	4	4-5	5	4	4-5	4	3-4	5	3-4	4	4-5	4
D <sub>11</sub>	4-5	4	4	3-4	4-5	4	4	3-4	3-4	4	4	3-4
D <sub>12</sub>	4	4-5	4-5	4	4	4-5	4	4	4	3-4	3-4	4

Abbreviations: (S) silk, (W) wool, and (C) cotton.

**Fastness to washing**

The test assessed using the launder-o-meter sponsored by the American Association of Textile Chemists and Colorists (A.A.T.C.C.). A test specimen (10 cm x 4 cm) of the dyed fiber was taken and samples (5 cm x 4 cm) of the white cotton and polyester fibers were placed in the container of the washing machine, with the necessary amount of soap solution (5 g/l)

A reflectance spectrophotometer was used for the colorimetric measurements on the dyed samples. K/S values given by the reflectance spectrophotometer are calculated at λ<sub>max</sub> and are directly correlated with the dye concentration on the substrate according to the Kubelka- Munk equation (Billmeyer and Saltzman, 1981):

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

Where, K is absorbance coefficient, S is scattering coefficient, and R is reflectance ratio.

The color coordinates (Table VI) indicates that the dyes have good affinity to silk, wool and cotton fibers. For silk fiber, Table VI showed that, the dyeing obtained using dye D<sub>12</sub> was lighter than other dyes, while dyeing obtained using dye D<sub>3</sub> was redder than other dyes. Dyeing obtained using dye D<sub>9</sub> was yellower and brighter than other dyes. Dyeing obtained using dye D<sub>1</sub> was darker and bluer than other dyes, while dyeing obtained using dye D<sub>12</sub> was greener and dye D<sub>5</sub> was duller than other dyes. Dye 5c having highest value of color strength (K/S) of 9.85. For wool fiber, Table VI showed that, the dyeing obtained using dye D<sub>10</sub> was lighter than other dyes, while dyeing obtained using dye D<sub>3</sub> was redder than other dyes and dye D<sub>9</sub> was brighter and yellower than other dyes. Dyeing obtained using dye D<sub>11</sub> was darker than other dyes, while dyeing obtained using dye D<sub>12</sub> was greener than other dyes. Dyeing obtained using dye D<sub>1</sub> was bluer and duller than other dyes. Dye D<sub>11</sub> having highest value of color strength (K/S) of 13.85. For cotton fiber, Table VI showed that, the dyeing obtained using dye D<sub>7</sub> was lighter than other dyes, while dyeing obtained using dye D<sub>2</sub> was redder and brighter than other dyes. Dyeing obtained using dye D<sub>10</sub> was yellower than other dyes. Dyeing obtained using dye D<sub>2</sub> was darker, D<sub>5</sub> was greener and duller than other dyes, while dyeing obtained using dye D<sub>1</sub> was bluer than other dyes. Dye D<sub>2</sub> having highest value of color strength (K/S) of 9.02.

**Table VI** Colorimetric (CIE lab) data of dyes D<sub>1</sub>-D<sub>12</sub> on silk, wool and cotton fibers

Dye No.	L*			a*			b*			C*			H*			K/S		
	S	W	C	S	W	C	S	W	C	S	W	C	S	W	C	S	W	C
D <sub>1</sub>	34.50	35.90	47.07	17.16	13.88	8.63	-2.01	-1.41	-9.51	17.28	13.95	12.84	353.33	354.19	312.21	7.12	6.06	2.67
D <sub>2</sub>	46.23	42.69	36.46	30.80	31.35	30.97	23.09	18.98	8.71	38.50	36.65	32.17	36.86	31.19	15.71	6.32	7.59	9.02
D <sub>3</sub>	51.80	46.56	64.76	32.98	32.69	20.87	21.87	16.41	5.59	39.57	36.58	21.60	33.56	26.65	14.99	4.98	5.89	1.06
D <sub>4</sub>	51.93	44.58	65.48	31.24	28.36	18.22	25.78	19.56	0.09	40.51	34.45	18.22	39.53	34.59	0.28	4.99	6.42	0.89
D <sub>5</sub>	50.23	49.49	63.47	5.80	8.18	0.35	6.61	12.26	3.25	8.79	14.74	3.27	48.76	56.30	83.84	2.84	4.04	0.95
D <sub>6</sub>	67.44	65.64	76.01	18.52	20.07	17.98	26.82	18.78	0.94	32.59	27.49	18.01	55.38	43.10	3.00	2.78	1.91	0.39
D <sub>7</sub>	69.87	59.08	86.44	11.97	21.51	5.69	17.07	19.01	2.45	20.85	28.70	6.19	54.96	41.47	23.26	1.48	3.26	0.10
D <sub>8</sub>	67.40	63.79	75.87	18.14	23.74	16.74	32.23	20.41	9.48	36.98	31.30	19.24	60.63	40.69	29.54	4.11	2.11	0.46
D <sub>9</sub>	61.21	52.26	85.12	24.83	29.84	4.45	48.71	44.83	2.88	54.67	53.85	5.30	62.99	56.35	32.95	8.24	11.85	0.13
D <sub>10</sub>	70.13	66.44	81.86	6.43	6.16	4.63	18.00	20.22	13.51	19.11	21.14	14.28	70.34	73.06	71.10	1.38	2.10	0.45
D <sub>11</sub>	37.12	31.63	60.90	18.52	17.62	10.89	14.98	11.51	2.91	23.82	21.04	11.28	38.97	33.15	14.97	9.85	13.85	1.10
D <sub>12</sub>	75.37	66.21	86.42	5.68	5.82	3.55	17.75	19.77	7.75	18.63	20.61	8.53	72.25	73.58	65.40	1.06	2.09	0.22

Abbreviations: (S) silk, (W) wool, and (C) cotton. Light fastness: (1) poor, (2) slight, (3) moderate, (4) fair, (5) good, and (6) very good, and Wash & Rubbing fastness: (1) poor, (2) fair, (3) good, (4) very good, and (5) excellent.

**Table VII** %Exhaustion and %fixation data of reactive dyes D<sub>1</sub>-D<sub>12</sub>

Dye No.	% Exhaustion			% Fixation		
	S	W	C	S	W	C
D <sub>1</sub>	70.85	67.80	69.52	92.67	90.38	84.58
D <sub>2</sub>	68.93	64.06	66.40	91.71	90.46	84.86
D <sub>3</sub>	67.55	62.91	66.16	91.40	89.85	83.69
D <sub>4</sub>	70.30	60.33	64.48	92.01	88.98	83.59
D <sub>5</sub>	70.58	60.04	64.96	90.95	90.19	83.73
D <sub>6</sub>	67.00	61.48	64.72	92.88	91.18	83.28
D <sub>7</sub>	66.18	60.33	64.48	91.82	90.55	83.59
D <sub>8</sub>	68.10	60.61	64.72	92.10	90.91	84.04
D <sub>9</sub>	67.28	58.89	64.72	91.77	91.15	83.28
D <sub>10</sub>	65.90	64.35	64.96	89.23	90.79	83.73
D <sub>11</sub>	64.53	71.54	69.28	88.85	90.97	87.70
D <sub>12</sub>	67.83	68.95	73.36	90.31	89.56	88.17

### Exhaustion and Fixation Study of Dyes

Data on percentage exhaustion and fixation were calculated by described method (Maradiya, 2002; Patel and Patel, 2005) and are summarized in Table VII. The percentage exhaustion value of dyes D<sub>1</sub>-D<sub>12</sub> for silk fiber varied from 64-70 %, for wool fiber varied from 58-71 % and for cotton fiber varied from 64-73 %. The percentage fixation of dyes D<sub>1</sub>-D<sub>12</sub> for silk fiber varied from 88-93 %, for wool fiber varied from 88-91 % and for cotton fiber varied from 83-88 %. Here exhaustion and fixation data on silk and wool fiber were higher than cotton fiber, due to the lower substantivity of the dyes with cotton fiber.

### Fastness Properties

The light fastness was assessed in accordance with BS: (1006-1978). The rubbing fastness test was carried out with Crock meter (Atlas) in accordance with the (AATCC- 1961) and the wash fastness test in accordance with (IS: 765-1979).

All the reactive dyes 7a-j show generally moderate to very good light fastness property and good to excellent washing and rubbing fastness properties on silk, wool, and cotton fibers. This is attributed to good penetration and affinity of the reactive dyes to the fibers. The high light fastness may be due to the higher attraction between dye and fiber and higher stability of resonance in the conjugated system. The fastness properties data of all the dyes 7a-j are summarized in Table V.

## CONCLUSION

A series of new water soluble reactive dyes derived from Quinazolin-4(3*H*)-one Moiety were synthesized in good yield. The synthesized dyes showed good to very good with respect to washing, rubbing and light fastness. The presence of azo group at the 5 position have a little influence on color coordinates (L\*, a\*, b\* and K/S).

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