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# Synthesis, Characterization and Application of Quinazolinone Based Reactive Dyes for Various Fibers

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**Abstract:** Seven hot brand heterocyclic mono azo reactive dyes (**7a-g**) have been synthesized by coupling diazotized 2-phenyl-3{4'-[(4"-aminophenyl)sulphonyl]phenyl}-quinazoline-4(3H)-one-6-sulphonic acid (**4**) with various 2-chloro-4-nitro anilino cyanurated coupling components (**6a-g**) and their dyeing performance on silk, wool, and cotton has been assessed. The purity of dyes was checked by thin layer chromatography. These dyes were identified by recording IR and $^1$H-NMR spectra. The $\lambda_{max}$, $R_f$ value, %exhaustion, %fixation, light fastness, wash fastness, rubbing fastness, reflectance (%$R$) value, and $K/S$ value have also been studied.

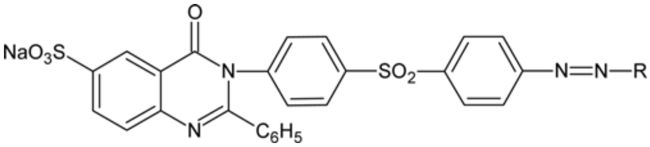
**Keywords:** 2-Phenyl-3[4'-(4"-aminophenyl sulphone)] phenyl-quinazoline-4(3H)-one-6-sulphonic acid, Monoazo reactive dyes, Silk, Wool, Cotton

## Introduction

The development of new structure of reactive dyes has been a subject of interest and many novel structures of these dyes useful in commercial application to silk, wool, and cotton as well as their blends with other fibers have been discovered.

The dyes containing heterocyclic moiety like quinazoline have been found to give a wide range of color shade with very good depth and levelness on each fibers, also shows excellent brightness and fastness properties including low sublimation and high thermal stability [1]. The largest group of quinazoline compounds includes 4-hydroxy quinazolines, 3,4-dihydroxyl-4-oxo-quinazolines, and 4(3H)-quinazolinones. These quinazolinone nucleus is the key component of a number of colored products [2].

A number of yellow to violet shades from 3H-2-(4'-chlorostyryl)-6-amino-4-oxoquinazoline have been reported [3]. Patel *et al*. have synthesized azo dyes containing 4-oxo-quinazoline dyes as a diazo component, these dyes were used to dye silk, wool, and viscose rayon with very good exhaustion and excellent properties [4]. Bhatti *et al*. [5] and Azaam *et al*. [6] synthesized quinazolinone based dyes and their fastness properties have been evaluated.



R=various 2-chloro-4-nitro anilino cyanurated coupling components (**6a-g**) (Table 1)

**Figure 1.** General structure of reactive dyes (**7a-g**).

In the present investigation, we wish to synthesize reactive dyes with quinazolinone structure. The different coupling components were selected to get a variety of shades. The dyeing performance of these dyes was examined on silk, wool, and cotton fibers, and also examined fastness properties and exhaustion study of these types of reactive dyes. The general structure of these reactive dyes is shown in Figure 1.

## Experimental

### Materials and Methods

All the used raw materials were of commercial grade and were further purified by recrystalization and redistillation before use. All melting points (m.p.) are uncorrected and expressed in $^o$C. The $R_f$ values [7] of all the dyes have been checked by thin layer chromatography and they are presented in Table 2. IR spectra were recorded on a Perkin-Elmer model-377 spectrophotometer instrument using KBr pellet method and $^1$H-NMR spectra on Brucker advance II 400 NMR spectrophotometer using TMS as internal standard and DMSO as solvent. The absorption spectra of all the dyes were recorded on a Beckman DB-GT grafting spectrophotometer. C, H, and O contents of dyes sample were estimated using C, H, and O analyzer, Carlo Erba, Italy. The light fastness was assessed in accordance with BS: 1006-1978 [8]. The rubbing fastness test was carried out with a crockmeter (Atlas) in accordance with AATCC-1961 [9] and the wash fastness test in accordance with IS: 765-1979 [10]. The %$R$ value has been calculated on a reflectance spectrophotometer (Datacolor SF600).

### Synthesis of Dyes

The synthesis of 2-phenyl-4-oxo-3,1-benzoxazine-6-suphonic acid (**1**) has been described in the previous work [11]. The

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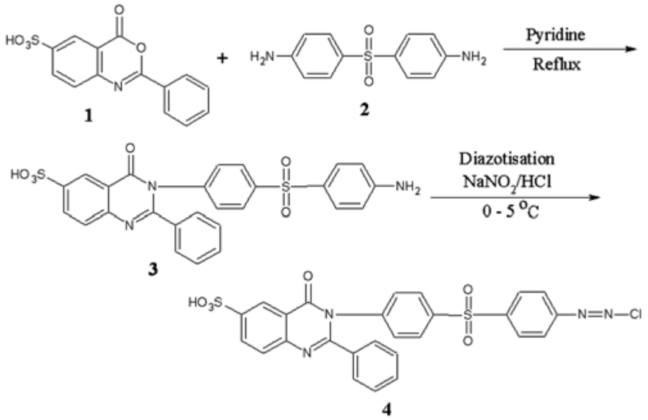
**Table 1.** Characterization data of intermediates (**1** to **3**) and reactive dyes (**7a-g**)

| Int./ Dyes | 2-chloro-4-nitro anilino cyanurated coupling component (R) | Molecular formula | Mol. weight | m.p. (°C) | Yield (%) | Elemental analysis | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | %C Found (Req.) | %H Found (Req.) | %N Found (Req.) |
| **1** | - | $C_{14}H_9O_5NS$ | 303 | 107 | 85.4 | 55.35<br>(55.44) | 2.82<br>(2.99) | 4.55<br>(4.62) |
| **2** | - | $C_{12}H_{12}O_2N_2S$ | 248 | 170 | 78.5 | 57.90<br>(58.05) | 4.75<br>(4.87) | 11.15<br>(11.28) |
| **3** | - | $C_{26}H_{19}O_6N_3S_2$ | 533 | 132 | 75.2 | 58.45<br>(58.53) | 3.47<br>(3.59) | 7.78<br>(7.88) |
| **7a** | H-acid (**6a**) | $C_{45}H_{25}O_{15}N_{10}Cl_2S_4Na_3$ | 1214 | - | 82.6 | 44.42<br>(44.53) | 1.98<br>(2.08) | 11.48<br>(11.54) |
| **7b** | Gamma acid (**6b**) | $C_{45}H_{26}O_{12}N_{10}Cl_2S_3Na_2$ | 1112 | - | 80.4 | 48.54<br>(48.61) | 2.28<br>(2.36) | 12.52<br>(12.60) |
| **7c** | J-acid (**6c**) | $C_{45}H_{26}O_{12}N_{10}Cl_2S_3Na_2$ | 1112 | - | 78.7 | 48.55<br>(48.61) | 2.30<br>(2.36) | 12.51<br>(12.60) |
| **7d** | N-methyl-J-acid (**6d**) | $C_{46}H_{28}O_{12}N_{10}Cl_2S_3Na_2$ | 1126 | - | 76.2 | 48.98<br>(49.07) | 2.44<br>(2.51) | 12.38<br>(12.44) |
| **7e** | N-phenyl- J-acid (**6e**) | $C_{51}H_{30}O_{12}N_{10}Cl_2S_3Na_2$ | 1188 | - | 82.4 | 51.50<br>(51.56) | 2.47<br>(2.55) | 11.70<br>(11.79) |
| **7f** | Chicago acid (**6f**) | $C_{45}H_{25}O_{15}N_{10}Cl_2S_4Na_3$ | 1214 | - | 83.2 | 44.45<br>(44.53) | 2.01<br>(2.08) | 11.46<br>(11.54) |
| **7g** | Laurant acid (**6g**) | $C_{45}H_{26}O_{11}N_{10}Cl_2S_3Na_2$ | 1096 | - | 75.8 | 49.21<br>(49.32) | 2.29<br>(2.39) | 12.70<br>(12.78) |

diaminodiphenylsulphone (**2**) compound was synthesized by the method reported in the literature [12]. The 2-phenyl-3-[4'-(4"-aminophenylsulphonyl)]-phenyl-4(3H)-quinazolinone-6-sulphonic acid (**3**) compound was synthesized by the method reported in the literature [13]. The mixture of 2-phenyl-4-oxo-3,1-benzoxazine-6-suphonic acid (**1**) (0.05 mole) and 4,4'-diaminodiphenylsulfone (**2**) (0.05 mole) in dry pyridine (50 m*l*) was heated under reflux for 6 h under anhydrous reaction condition and then allowed to cool at room temperature. The reaction mixture was treated with dil. HCl and stirred. A solid, which was precipitated out, was filtered off and washed with water to remove any adhered pyridine. The crude quinazoline thus obtained (**3**) was dried under vacuum and recrystallized from ethanol (Scheme 1).

The 2-phenyl-3-[4'-(4"-aminophenylsulfone)]-phenyl-4(3H)-quinazolinone-6-sulfonic acid (2.58 g, 0.005 mole) (**3**) was suspended in water (60 m*l*). Hydrochloric acid (0.36 g) was added drop wise to this well stirred suspension. The mixture was gradually heated up to 70 °C, till clear solution was obtained. The solution was cooled at 0-5 °C in an iced water bath. A solution of $NaNO_2$ (0.6 g) in water (4 m*l*) previously cooled to 0 °C, was then added over a period of 5 min with stirring. The stirring was continued for 1 h, maintaining the same temperature, with positive test for nitrous acid on starch iodide paper. After just destroying excess of nitrous acid with required amount of a solution of sulphamic acid, the clear diazo solution (**4**), which would be used for subsequent coupling reaction, was obtained (Scheme 1).

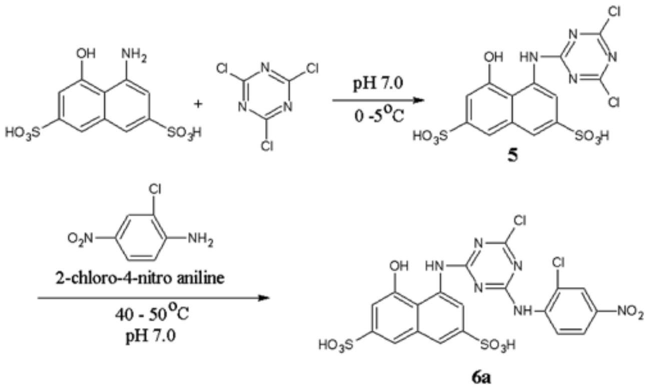
Cyanuric chloride (1.85 g, 0.01 mole) was stirred in acetone (25 m*l*) at a temperature below 5 °C for a period of 1 h. A neutral solution of H-acid (3.19 g, 0.01 mole) in an aqueous sodium carbonate solution (10 % w/v) was then added in small lots in about 1 h. The pH was maintained neutral by simultaneous addition of sodium carbonate solution (1 % w/v). The reaction mass was stirred at 0-5 °C for further 4 h when a clear solution was obtained (**5**) (Scheme 2). The resultant solution was used for condensation reaction with 4-chloro aniline.



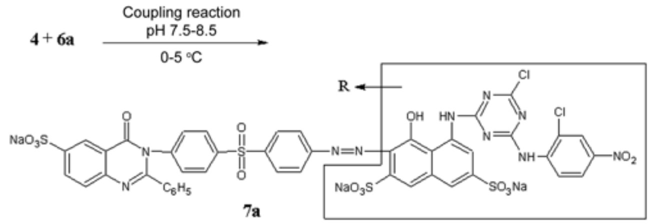
**Scheme 1.** The route of the synthesis of 2-phenyl-3-[4'-(4"-aminophenylsulphonyl)]-phenyl-4(3H)-quinazolinone-6-sulphonic acid (**3**) and its diazonium salt (**4**).

The temperature of ice-cooled well stirred solution of cyanurated H-acid (**5**) was gradually raised to 45 °C for half an hour. To this cyanurated H-acid, 2-chloro-4-nitro aniline (1.73 g, 0.01 mole) was added drop wise at same temperature, during a period of 30 min, maintaining the pH neutral by simultaneous addition of sodium bicarbonate (1 % w/v). After the addition was completed, stirring was continued for further 3 h. The 2-chloro-4-nitro anilino cyanurated H-acid solution (**6a**) (Scheme 2) thus obtained was subsequently used for further coupling reaction.

To an iced cold and stirred solution of 2-chloro-4-nitro anilino cyanurated H-acid (6.03 g, 0.01 mole) (**6a**), a freshly prepared diazo solution (2.91 g, 0.005 mole) (**4**) was added drop wise over a period of 10-15 min. The pH was maintained at 7.5 to 8.5 by simultaneous addition of sodium carbonate solution (10 % w/v). During coupling, the purple solution was formed. The stirring was continued for 3-4 h, maintaining the temperature below 5 °C. The reaction mixture was heated up to 60 °C and sodium chloride (12 g) added until the coloring material was precipitated. It was stirred for 1 h, filtered, and washed with a small amount of sodium chloride solution (5 % w/v). The solid was dried at 80-90 °C and extracted with DMF (Scheme 3). The dye was precipitated by diluting the DMF-extract with an excess of chloroform. A violet dye was then filtered, washed with chloroform, and dried at 60 °C (Scheme 3).



**Scheme 2.** The route of the synthesis of the Cyanurated H-acid **5** and 2-chloro-4-nitro anilino cyanurated H-acid **6a**.



where R=various 2-chloro-4-nitro aniline cyanurated coupling components (**6a-g**).

**Scheme 3.** The route of the synthesis of the Dye **7a**.

Following the above procedure, other reactive dyes **7b-g** were synthesized using 2-chloro-4-nitro anilino cyanurated coupling components such as Gamma acid (**6b**), J-acid (**6c**), N-methyl-J-acid (**6d**), N-phenyl-J-acid (**6e**), Chicago acid (**6f**), and Laurant acid (**6g**) (Table 1).

## Results and Discussion

A series of reactive dyes (**7a-g**) were prepared by diazotization of 2-phenyl-3-[4'-(4"-aminophenylsulphonyl)]-phenyl-4(3H)-quinazolinone-6-sulphonic acid (**3**) with various 2-chloro-4-nitro anilino cyanurated coupling components (**6a-g**) (Table 1). Diazotization was carried out using sodium nitrite and hydrochloric acid and the coupling was done in a moderately alkaline medium at 0-5 °C. The dyes were characterized by their infrared spectrum [14,15]. All the dyes showed characteristic band at 3350-3770 $cm^{-1}$ for O-H and N-H stretching vibrations, 1605-1620 $cm^{-1}$ for N=N stretching vibrations, 1665-1675 $cm^{-1}$ for C=O stretching vibrations, 1380-1390 $cm^{-1}$ for C-N stretching vibrations, and 770-780 $cm^{-1}$ for C-Cl stretching vibrations. All IR and $^1$H-NMR data of intermediates (**1** to **3**) and dyes (**7a-g**) are summarized in Table 2.

### Dyeing Properties

All the **7a-g** were applied on silk, wool, and cotton fabrics in 2 % shade according to the usual procedure [16]. Dyeing was carried out at pH 8.0-9.0 on cotton while on silk and wool pH was adjusted to 3.0-4.0 by dilute acetic acid and formic acid. The dyes cover almost all the visible range and give generally yellow to purple hues. The variations in the hues of the dyed fabric result from the alteration in the coupling components.

### Wash-off Process

In this aspect of our study, the dyed fabrics 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. Most of the hydrolyzed dye having low substantivity was released easily from the substrate after two or three washes. Also higher molecular size greatly increases the wash fastness.

### Spectral Properties

The absorption maxima ($\lambda_{max}$) and logarithm of the molar extinction co-efficient (log ε) of the dyes **7a-g** are given in Table 3. The absorption maxima of **7a-g** were determined in water at 28 °C. The absorption maxima were in the range of 430-520 nm. The value of logarithm of molar extinction co-efficient (log ε) of the dyes **7a-g** were in the range 4.15-4.48, indicating their good absorption intensity.

**Table 2.** IR and $^1$H-NMR data of intermediates (**1** to **3**) and dyes (**7a-g**)

| Int./ Dyes | IR (KBr, cm$^{-1}$) | $^1$H-NMR Chemical shift (DMSO-d$_6$), δ, ppm |
|---|---|---|
| **1** | 3025 (C-H), 1725 (C=O), 1182 (C-O), 1605 (C=N), 1345, 1160 (S=O, asym. & sym.) | 10.8 (1H, s, $SO_3H$), 6.75-8.04 (8H, m, Ar-H) |
| **2** | 3455 (N-H), 3030 (C-H), 1355, 1130 (S=O, asym. & sym.) | 2.45 (4H, s, $NH_2$), 6.68-7.95 (8H, m, Ar-H) |
| **3** | 3450 (N-H), 3035 (C-H), 1690 (C=O), 1608 (C=N), 1355, 1135 (S=O, asym. & sym.) | 2.38 (2H, s, $NH_2$), 10.82 (1H, s, $SO_3H$), 6.72-8.10 (16H, m, Ar-H) |
| **7a** | 3375-3755 (O-H & N-H), 1675 (C=O), 1615 (N=N), 1385 (C-N), 1490, 1325 (N=O, asym. & sym.), 1365, 1135, 1040 (S=O), 772 (C-Cl) | 3.35 (1H, s, OH), 4.10 (2H, s, NH), 6.75-8.20 (22H, m, ArH). |
| **7b** | 3360-3750 (O-H & N-H), 1670 (C=O), 1618 (N=N), 1382(C-N), 1495, 1320 (N=O, asym. and sym.), 1362, 1137, 1042 (S=O), 775 (C-Cl) | 3.32 (1H, s, OH), 4.15 (2H, s, NH), 6.72-8.25 (23H, m, ArH) |
| **7c** | 3370-3750 (O-H & N-H), 1665 (C=O), 1612 (N=N), 1390 (C-N), 1491, 1323 (N=O, asym. and sym.), 1360, 1131, 1037 (S=O), 770 (C-Cl) | 3.30 (1H, s, OH), 4.20 (2H, s, NH), 6.75-8.25 (23H, m, ArH). |
| **7d** | 3360-3765 (O-H & N-H), 1670 (C=O), 1610 (N=N), 1380 (C-N), 1492, 1320 (N=O, asym. and sym.), 1360, 1140, 1045 (S=O), 760 (C-Cl) | 3.31 (1H, s, OH), 2.51 (3H, s, N-$CH_3$), 4.02 (1H, s, NH), 6.75-8.04 (23H, m, ArH) |
| **7e** | 3355-3760 (O-H & N-H), 1665 (C=O), 1615 (N=N), 1375 (C-N), 1490, 1325 (N=O, asym. and sym.), 1362, 1145, 1040 (S=O), 765 (C-Cl) | 3.28 (1H, s, OH), 4.08 (1H, s, NH), 6.68-8.20 (28H, m, ArH) |
| **7f** | 3370-3765 (O-H & N-H), 1672 (C=O), 1618 (N=N), 1385(C-N), 1495, 1320 (N=O, asym. and sym.), 1360, 1140, 1042 (S=O), 770 (C-Cl) | 3.30 (1H, s, OH), 4.12 (2H, s, NH), 6.72-8.30 (22H, m, ArH) |
| **7g** | 3360-3770 (O-H & N-H), 1668 (C=O), 1605 (N=N), 1380 (C-N), 1490, 1325 (N=O, asym. and sym.), 1365, 1142, 1040 (S=O), 775 (C-Cl) | 4.12 (2H, s, NH), 6.72-8.28 (24H, m, ArH) |

Abbreviation: s-singlet, d-doublet, m-multiplet.

**Table 3.** $R_f$ value, absorption maxima, logarithm of molar extinction coefficient, reflectance and *K/S* value of reactive dyes (**7a-g**)

| Dyes | $R_f^a$ value | $\lambda_{max}^b$ nm | Log ε | Reflectance (%*R*) | *K/S* |
|---|---|---|---|---|---|
| **7a** | 0.44 | 520 | 4.48 | 20.54 | 1.4860 |
| **7b** | 0.42 | 455 | 3.28 | 63.95 | 0.1013 |
| **7c** | 0.36 | 460 | 4.30 | 35.03 | 0.6036 |
| **7d** | 0.38 | 482 | 4.15 | 46.82 | 0.2988 |
| **7e** | 0.45 | 495 | 4.22 | 31.98 | 0.7225 |
| **7f** | 0.40 | 510 | 4.36 | 45.62 | 0.3241 |
| **7g** | 0.37 | 445 | 4.32 | 34.61 | 0.6177 |

[a]Determined by TLC using benzyl alcohol+DMF+water (30:20:20) solvent system on Silica gel G $F_{254}$ TLC plate, [b]determined in water at 28 °C at $2\times10^{-3}$ M dye concentration.

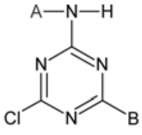
Some substances appear colored due to the presence of such functional groups like C=C, C=O, N=N, etc. which are capable of absorbing visible and/or ultraviolet radiation (800-200 nm). Such functional groups, which confer color on substances, are known as chromophores. And an auxochrome is a functional group which does not absorb radiation longer than 200 nm, but when attached to a given chromophore, causes a shift in absorption to longer wavelength and also increases the depth of absorption.

Dyes **7a-g** consist of many chromophoric groups like C=C, C=O, N=N, $NO_2$, C=N, etc. These chromophores are capable of absorbing visible and/or UV radiation (800-200 nm) to exhibit a color.

With comparison of dyes **7a-g**, dyes **7a** and **7f** have $\lambda_{max}$ greater than **7g** because **7a** and **7f** having hydroxyl group (auxochrome) which extends the conjugation due to the lone pair of electrons on the oxygen atom. Also it is found that the chromophoric group like N=N is attached ortho to the -OH group, causing resonance effects, which also increases the $\lambda_{max}$ value. Another comparison of dyes **7a** with **7b** and **7c** shows that dye **7a** possesses higher $\lambda_{max}$ value due to the fact that dye **7a** possess additional auxochrome like sulfonic acid group. The same effect is produced in the comparison of dyes **7a** with **7d** and **7e**.

From the data reported in Table 3, it is apparent that the value of $\lambda_{max}$ depends on the coupling components used. The color change observed for each dye is due to the oscillation of electrons and the presence of additional substituents. In **7a** there is more place for the oscillation of electrons and also the presence of both -OH and -NH- groups, thus $\lambda_{max}$ = 520 nm. In **7b**, **7c**, **7d**, **7e**, and **7g** slight lower effect is produced due to the less number of electrons and thus the oscillation is fast. Therefore $\lambda_{max}$ of all these dyes are shorter than **7a**. For dye **7f**, the substituent is same as in **7a**, but the oscillation of electron is fast due to the vicinity of -NH- and -$SO_3$Na group, i.e., neutralization of electrons takes place rapidly ($\lambda_{max}$=510 nm).

A bathochromic shift was observed, the magnitude of which depended on the nature of substituents at the terminal amino group. The following trend was observed in J-acid, N-methyl-J-acid, and N-phenyl-J-acid.



**7c** ($\lambda_{max}$ = 460 nm)

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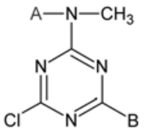

**7d** ($\lambda_{max}$ = 482 nm)

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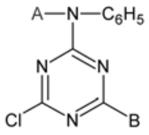

**7e** ($\lambda_{max}$ = 495nm)

Where A=Quinazolinone molecule and
B=2-Chloro-4-nitro aniline molecule

Dye molecule **7c** shows $\lambda_{max}$ 460 nm and **7d** shows $\lambda_{max}$ 482 nm. Thus, there is a bathochromic shift of 22 nm in the $\lambda_{max}$ of **7c** due to the presence of auxochrome $CH_3$. Similarly compound **7e** shows $\lambda_{max}$ 495 nm; thus, there is a bathochromic shift of 13 nm in the $\lambda_{max}$ of **7d** due to the presence of $C_6H_5$ ring which increases the $\lambda_{max}$ by extending the conjugation through resonance. The combination of chromophore and auxochrome behaves as a new chromophore which extends the conjugation involving the lone pair of electrons on the nitrogen atom resulting in the increase value of $\lambda_{max}$.

### Reflectance Measurements

A reflectance spectrophotometer (Datacolor SF600) was used for the colorimetric measurements on the dyed samples. *K/S* values given by the reflectance spectrophotometer are calculated at $\lambda_{max}$ and are directly correlated with the dye concentration on the dye substrate according to the Kubelka-Munk equation [17]: $K/S=(1-R)^2/2R$, where $K$=absorbance coefficients, $S$=scattering coefficients, and $R$=reflectance ratio. From the Table 3 it is apparent that dye **7a** contains maximum value of *K/S* and minimum value of reflectance (Figure 2 shows graph of reflectance vs wavelength).

### Exhaustion and Fixation Properties

The data of percentage exhaustion of 2 % dyeing on cotton range from 65-74 %, while silk fabric ranges from 67-75 % and wool ranges from 65-71 %. The percentage fixation of 2 % dyeing on cotton fabric ranges from 84-91 %, whereas silk ranges from 85-93 % and wool ranges from 83-93 % (Table 4). The higher percentage exhaustion on the fibers is to be expected due to its relatively open structure [18] (Figures 3 and 4).

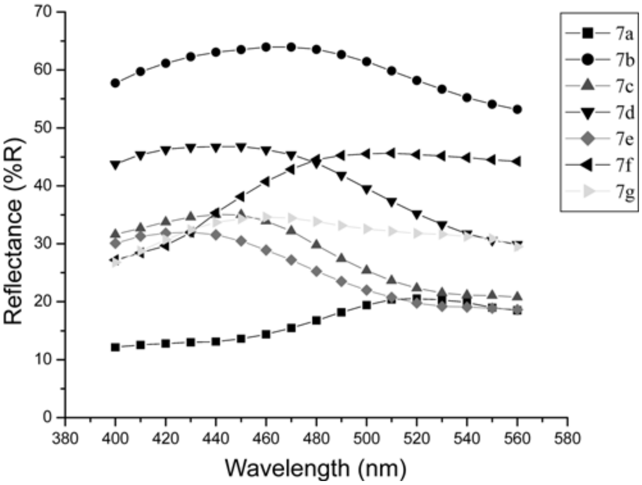

**Figure 2.** Plot of reflectance (%*R*) versus wavelength (nm) for dyes **7a-g**.

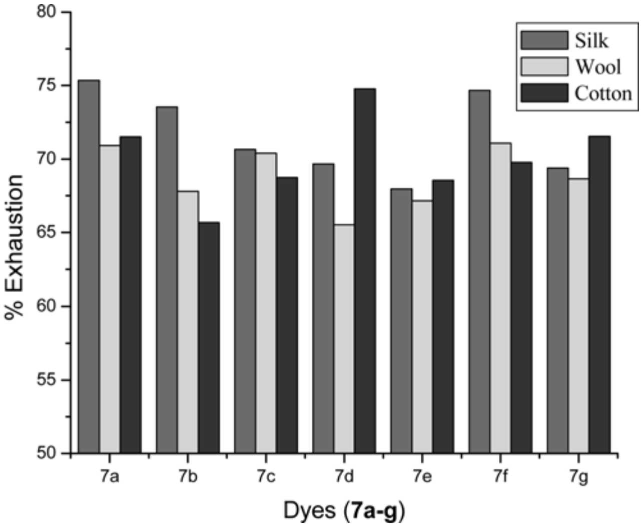

**Figure 3.** %Exhaustion of dyes **7a-g**.

**Table 4.** Result of exhaustion and fixation study of reactive dyes (**7a-g**) on silk, wool, and cotton

| Dyes | Color | % Exhaustion | | | % Fixation | | |
|---|---|---|---|---|---|---|---|
| | | S | W | C | S | W | C |
| **7a** | Pink | 75.35 | 70.92 | 71.50 | 91.60 | 93.10 | 91.55 |
| **7b** | Yellow | 73.55 | 67.80 | 65.67 | 88.45 | 89.50 | 83.95 |
| **7c** | Dark Yellow | 70.65 | 70.40 | 68.75 | 85.75 | 91.62 | 86.52 |
| **7d** | Orange | 69.66 | 65.52 | 74.78 | 90.14 | 86.75 | 88.96 |
| **7e** | Red | 67.95 | 67.15 | 68.55 | 92.95 | 83.48 | 86.58 |
| **7f** | Light Purple | 74.68 | 71.08 | 69.76 | 84.35 | 88.75 | 87.62 |
| **7g** | Light Yellow | 69.38 | 68.65 | 71.54 | 85.76 | 83.58 | 90.75 |

Abbreviation: S=Silk, W=Wool, C=Cotton.

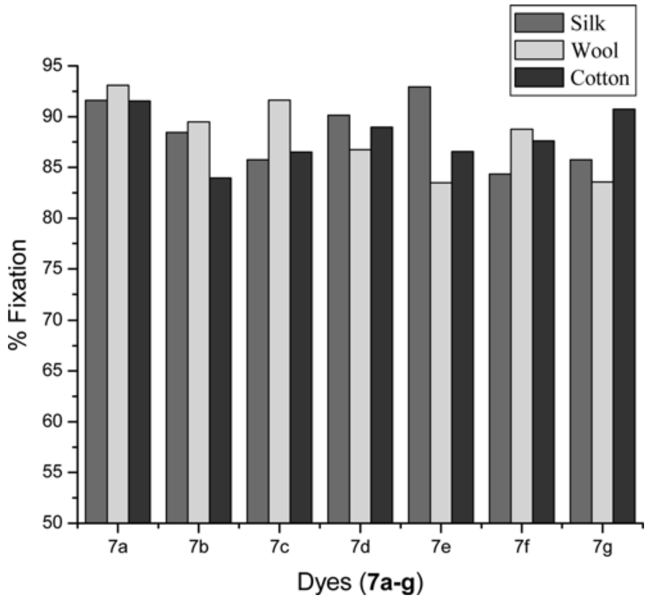

**Figure 4.** %Fixation of dyes **7a-g**.

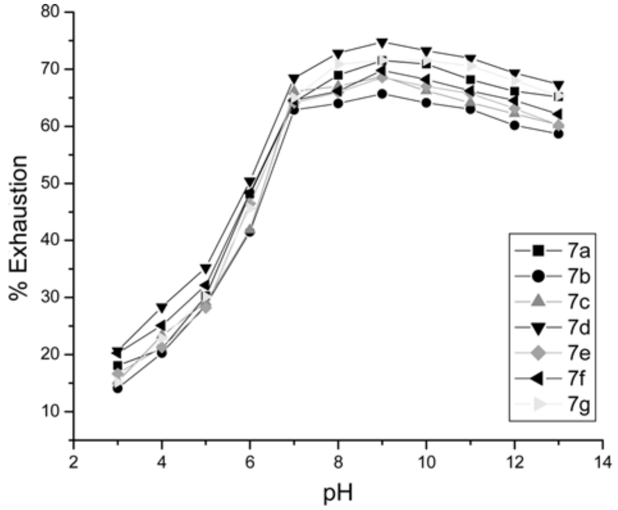

**Figure 5.** Effect of pH on exhaustion for cotton fiber.

## Effect of pH on Exhaustion and Fixation

From the Figures 5 and 6 it is apparent that exhaustion (%*E*) of the dyes (**7a-j**) on cotton fibers increased with increasing pH of application and that fixation (%*F*) of the dyes on cotton fibers increased with increasing pH of application. Result shows that the highest total fixation efficiency was achieved at pH 9.0.

For wool and silk fibers the exhaustion (%*E*) of the dyes (**7a-j**) increased with decreasing pH of the application and that's why fixation (%*F*) of the dyes on wool and silk fibers increased with increasing pH of application (Figures 7, 8, 9, and 10).

The result shows that the highest total fixation efficiency was achieved at pH 4.0 for silk and pH 5.0 for wool fibers.

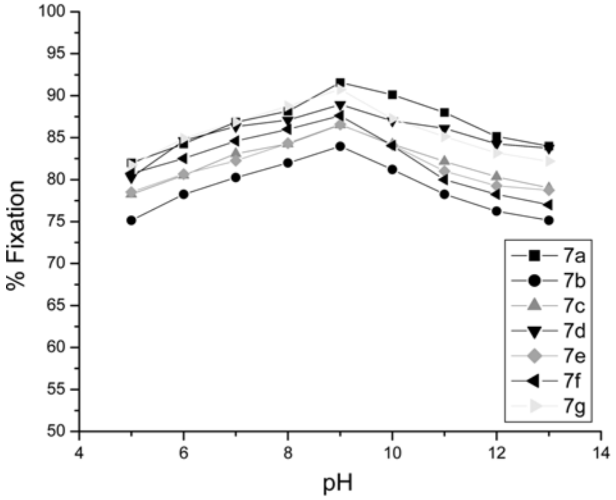

**Figure 6.** Effect of pH on fixation for cotton fiber.

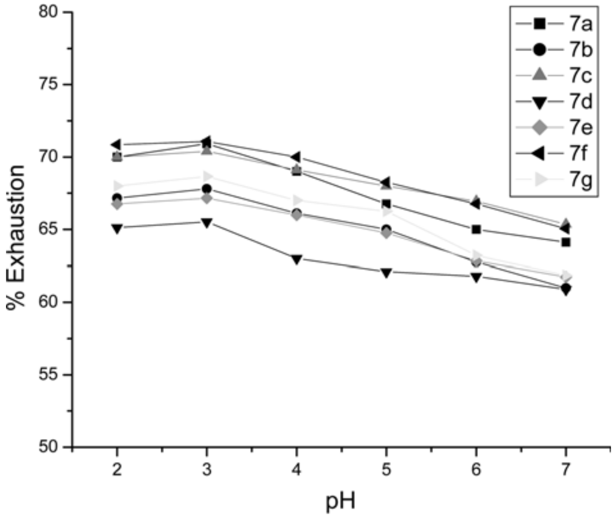

**Figure 7.** Effect of pH on exhaustion for wool fiber.

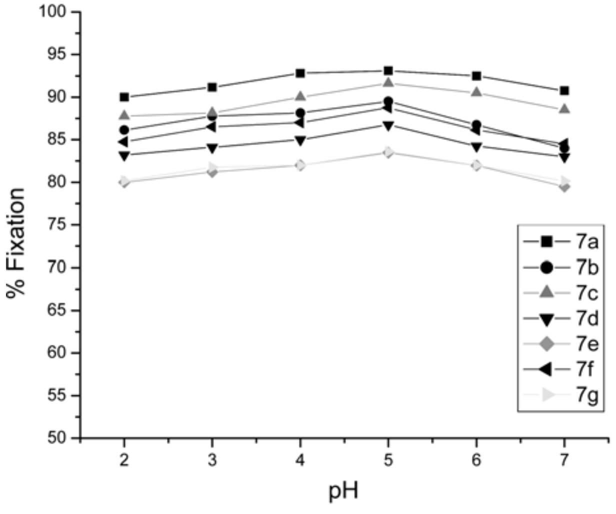

**Figure 8.** Effect of pH on fixation for wool fiber.

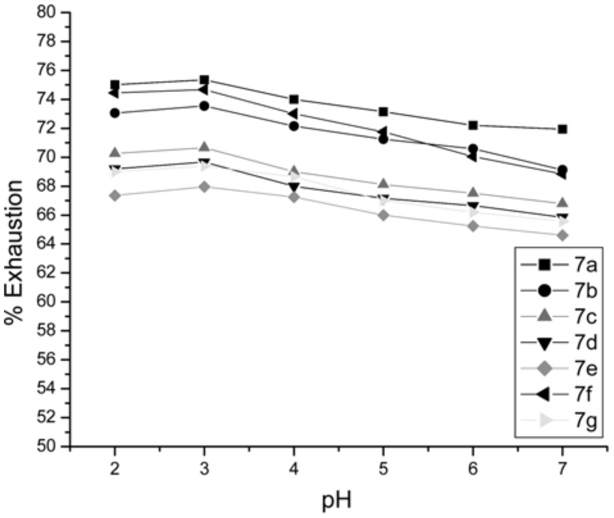

**Figure 9.** Effect of pH on exhaustion for silk fiber.

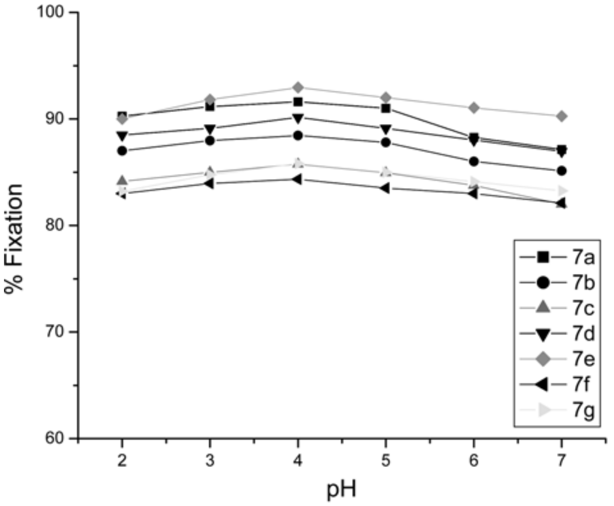

**Figure 10.** Effect of pH on fixation for silk fiber.

At lower pH values higher extent of protonation of amino groups within wool and silk fibers leads to higher exhaustion and low fixation, because the ensuing low concentration of nucleophilic amino groups in the substrates are unable to react with the dye. In addition at low pH, the dye is prevalent only at low concentration in the dye bath.

### Fastness Properties

All the dyes (**7a-g**) show generally moderate to good light fastness properties. This reveals that the dyes show a remarkable fading or photodegradation on dyed fibers. No proper generalization is possible with these a series of dyes. The washing and rubbing fastnesses for silk, wool, and cotton range from good to excellent (Table 5), this indicates the good penetration and affinity of these reactive dyes for the fibers and also indicates good substantivity of these dyes for the fibers.

## Conclusion

Reactive dyes based on 2-phenyl-3[4'-(4"-aminophenylsulphonyl)] phenyl-quinazoline-4(3H)-one-6-sulphonic acid were synthesized. These dyes gave mostly yellow, orange, red, and purple shades on silk, wool, and cotton fibers having very good to excellent fastness properties on the fabrics. The presence of quinazolinone structure in all the dyes gives high tinctorial power and excellent fastness properties and also such dyes gave excellent dyeing properties. The intrinsic conjugation in the quinazolinone structure results in excellent color strength. The heteroatoms in the dye structure results in bathochromaticity and leads to brightness of shades. The dyes have good exhaustion and fixation properties. This indicates that the dyes have good affinity and solubility with the fabric. And these dyes showed a remarkable degree of levelness after washing, which indicated good penetration and excellent affinity to the fibers. The introduction of a triazine group to the dye

**Table 5.** Fastness properties of reactive dyes (**7a-g**) on silk, wool and cotton

| Dyes | Light fastness | | | Wash fastness | | | Rubbing fastness | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Dry | | | Wet | | |
| | S | W | C | S | W | C | S | W | C | S | W | C |
| **7a** | 3 | 5 | 3 | 4-5 | 5 | 4 | 4-5 | 3 | 4-5 | 3-4 | 3 | 4 |
| **7b** | 5 | 4-5 | 5 | 5 | 3-4 | 5 | 4 | 3-4 | 3 | 4 | 5 | 3 |
| **7c** | 4-5 | 4 | 3-4 | 4-5 | 4 | 4 | 3 | 4 | 5 | 4-5 | 5 | 4 |
| **7d** | 5 | 4-5 | 4-5 | 4 | 4 | 3-4 | 4 | 5 | 4 | 3 | 5 | 4-5 |
| **7e** | 5 | 4 | 3-4 | 3-4 | 3 | 5 | 4-5 | 3-4 | 3 | 5 | 4-5 | 4 |
| **7f** | 4 | 3 | 4-5 | 4 | 5 | 3-4 | 4 | 5 | 4-5 | 4 | 4 | 5 |
| **7g** | 3-4 | 5 | 4 | 3 | 3-4 | 4 | 4-5 | 4 | 4 | 3-4 | 3 | 3-4 |

Abbreviation: S=Silk, W=Wool, C=Cotton, Light fastness: 1-poor, 2-slight, 3-moderate, 4-fair, 5-good, 6-very good, Wash & Rubbing fastness: 1-poor, 2-fair, 3-good, 4-very good, 5-excellent.

molecule improves the degree of exhaustion and fixation of the dyes.

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