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# Dyeing performance of heterocyclic monoazo dyes based on 3-amino 1*H*-pyrazolo[3,4-*b*]quinoline derivatives on various fibers

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## ABSTRACT

*1H-pyrazolo[3,4-*b*]quinolin-3-amine derivatives have been synthesized by the reaction between substituted phenyl pyrazolones and 3-amino-1H-pyrazolo [3,4-*b*] quinoline. The novel compound structures have been established on the basis of their substituted phenyl pyrazolones derivatives. All the azo compounds were characterized by their percentage yield, melting point, elemental analysis, UV visible spectra, IR spectra, NMR spectra and dyeing performance on nylon, wool, silk and polyester fibers. All the synthesized dyes gave moderate to excellent fastness properties on each fiber.*

**Keywords:** 3-amino-1*H*-pyrazolo[3,4-*b*]quinoline, phenyl pyrazolones, dyeing, fastness properties.

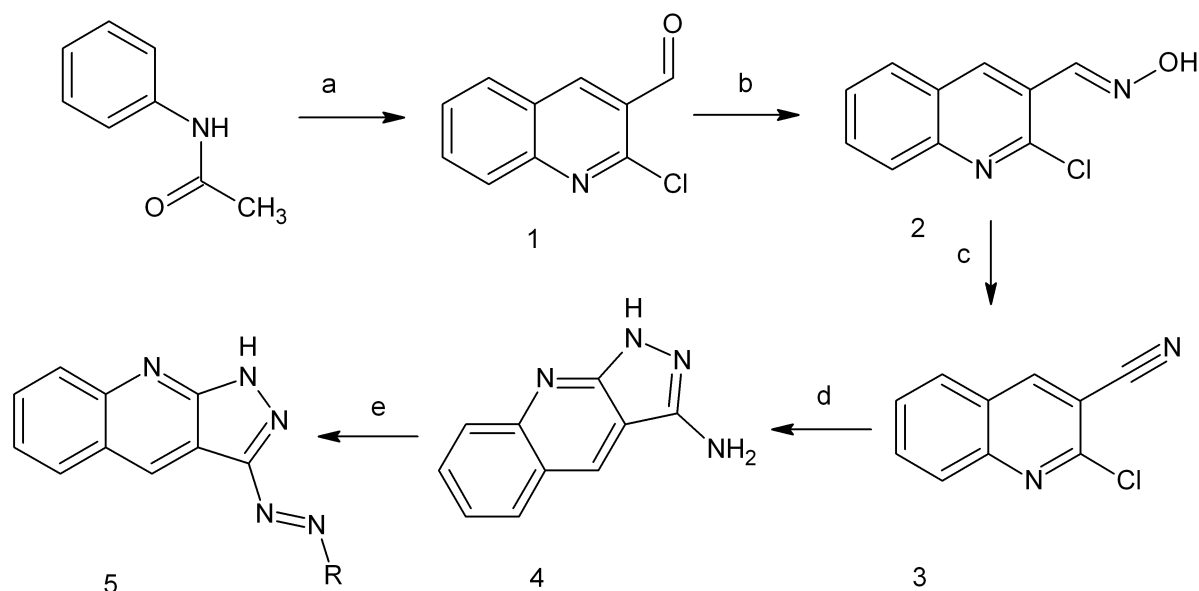
## INTRODUCTION

Various dyes based on heterocyclic system are recognized to possess tremendous fastness properties on various fibers. A number of azo dyes have been prepared from amino heterocycles [1]. The compound having heterocyclic system like quinolines, quinazoline and its derivatives are used as intermediates in dyestuff industry [2-10]. In the present endeavor, 3-amino-1*H*-pyrazolo[3,4-*b*]quinoline have been synthesized through coupling reaction between quinoline and pyrazolones to give a series of heterocyclic monoazo dyes having good fastness properties. All the azo compounds were also applied on silk, wool, nylon and polyester fibers as acid and dispersed dyes and their dyeing properties have been assessed. All the synthesized dyes exhibit moderate to good fastness properties on each fiber.

## MATERIALS AND METHODS

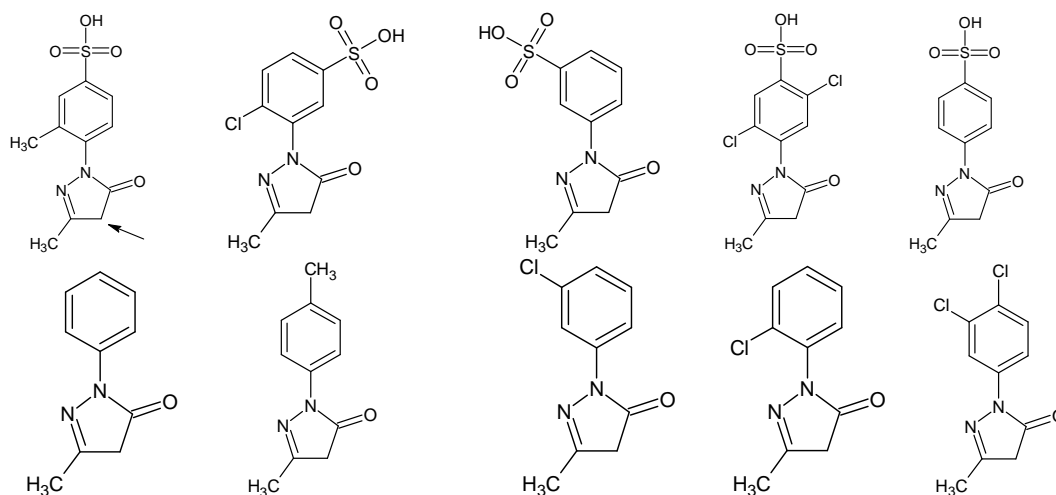
All chemicals were of analytical grade and used directly. All melting points were determined in PMP-DM scientific melting point apparatus and are uncorrected. The purity of all dyes was determined by thin-layer chromatography (TLC) [11] using silica gel-G coated Al-plates (0.5

mm thickness, Merck). Infrared spectra were recorded on a Shimadzu FT-IR 8400S model using KBr pellets. <sup>1</sup>H NMR spectra were acquired on a Varian 400 MHz model spectrophotometer using DMSO as a solvent and TMS as internal reference (chemical shifts in  $\delta$ , ppm). Elemental analysis of C, H and N were carried on Carlo Erba 1108 instrument. The light fastness was assessed in accordance with BS: 1006-1978 (Standard test method, 1978, 1994). The rubbing fastness test was carried out with a Crock meter (Atlas) in accordance with AATCC-1961 (AATCC test method, 1961) and the wash fastness test in accordance with IS: 765-1979 (Indian standard ISO, 1979).



**Scheme 1.** Reagents: a) DMF, POCl<sub>3</sub> (3: 12), reflux, 6 h, 80-90°C. b) NH<sub>2</sub>OH, CH<sub>3</sub>COONa, stirring RT, 1 h. c) SOCl<sub>2</sub>, benzene, reflux, 2 h. d) NH<sub>2</sub>NH<sub>2</sub>H<sub>2</sub>O, ethanol, e) NaNO<sub>2</sub>, HCl, 0- 5°C, diazotization & coupling

#### Various coupling components (R)



#### 2-chloroquinoline-3-carbaldehyde (1)

The title compound was synthesized following a reaction according to a procedure described in the literature. Yield 70%, m.p.146-152°C [12].

**2-chloro-3-quinoline-carboxaldehyde oxime (2)**

The title compound was synthesized following a reaction according to a procedure described in the literature. Yield 78%, m.p.238-242°C [13].

**2-chloro-3-quinoline carbonitrile (3)**

The title compound was synthesized following a reaction according to a procedure described in the literature. Yield 84%, m.p.190-205°C [14].

**1H-pyrazolo[3,4-b]quinolin-3-amine (4)**

The title compound was synthesized following a reaction according to a procedure described in the literature. Yield 79%, m.p.290-300°C [14].

**Diazotization and Coupling (5)**

Diazotization and coupling was carried out by usual procedure [15].

**Compound (R<sub>1</sub>):** Yield: 85%; m.p. >300°C (dec.); IR (KBr,cm<sup>-1</sup>) : 3198 cm<sup>-1</sup> (-NH-), 1491-1579 cm<sup>-1</sup> (-N=N-), 3032-3059 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2821-2997 cm<sup>-1</sup> (-C-H) stretching of methyl group; <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.69 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.91 (m, 8H, Ar-H), 13.51(s, 1H, -NH); Anal. Calcd. for C<sub>21</sub>H<sub>17</sub>N<sub>7</sub>O<sub>4</sub>S: C, 54.42%; H, 3.70%; N, 21.15%. found: C, 54.48%; H,3.80%; N,20.95%.

**Compound (R<sub>2</sub>):**Yield: 79%; m.p. >300°C (dec.); IR (KBr,cm<sup>-1</sup>) : 3178 cm<sup>-1</sup> (-NH-), 1595 cm<sup>-1</sup> (-N=N-), 3032-3057 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group, 723 cm<sup>-1</sup> (-C-Cl-); <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.42 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.90 (m, 8H, Ar-H), 13.70(s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>14</sub>ClN<sub>7</sub>O<sub>4</sub>S: C, 49.64%; H, 2.92%; N, 20.26%. found: C, 49.70%; H,2.96%; N, 20.30%.

**Compound (R<sub>3</sub>):**Yield: 88%%; m.p. >300°C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120 cm<sup>-1</sup> (-NH-), 1475-1618 cm<sup>-1</sup> (-N=N-), 3032-3057 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group; <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.35 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.91 (m, 9H, Ar-H), 13.65 (s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>15</sub>N<sub>7</sub>O<sub>4</sub>S: C, 53.45%; H, 3.36%; N, 21.82%. found: C, 53.41%; H, 3.32%; N, 21.72%.

**Compound (R<sub>4</sub>):** Yield: 90%; m.p. >300°C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120-3178 cm<sup>-1</sup> (-NH-), 1489-1585 cm<sup>-1</sup> (-N=N-), 3055-3084 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group, 742 cm<sup>-1</sup> (-C-Cl-); <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.91 (m, 7H, Ar-H), 13.51(s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>13</sub>Cl<sub>2</sub>N<sub>7</sub>O<sub>4</sub>S: C, 46.34%; H, 2.53%; N, 18.92%. found: C, 46.30%; H, 2.64%; N, 18.79%.

**Compound (R<sub>5</sub>):** Yield: 82%; m.p. >300°C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120-3178 cm<sup>-1</sup> (-NH-), 1494 cm<sup>-1</sup> (-N=N-), 3032-3055 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group.; <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.91 (m, 9H, Ar-H), 13.70 (s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>15</sub>N<sub>7</sub>O<sub>4</sub>S: C, 53.45%; H, 3.36%; N, 21.82%. found: C, 53.52%; H, 3.39%; N, 21.88%.

**Compound (R<sub>6</sub>):** Yield: 77%; m.p. >300<sup>0</sup>C (dec.); IR (KBr,cm<sup>-1</sup>) : 31230-3188 cm<sup>-1</sup> (-NH-), 1510-1572 cm<sup>-1</sup> (-N=N-), 3032-3043 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2810-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group; <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.44 (s, 3H, Me), 2.69 (s, 3H, Me), 2.85 (s, 1H, -CH), 7.43-8.91 (m, 9H, Ar-H), 13.51(s, 1H, -NH); Anal. Calcd. for C<sub>21</sub>H<sub>17</sub>N<sub>7</sub>O: C, 65.44%; H, 4.97%; N, 25.44%, found: C, 65.40%; H, 4.90%; N, 25.40%.

**Compound (R<sub>7</sub>):** Yield: 81%; m.p. >300<sup>0</sup>C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120-3178 cm<sup>-1</sup> (-NH-), 1541-1589 cm<sup>-1</sup> (-N=N-), 3032-3053 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2818-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group, 781 cm<sup>-1</sup> (-C-Cl-); <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85(s, 1H, -CH), 7.43-8.91 (m, 9H, Ar-H), 13.65 (s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>14</sub>ClN<sub>7</sub>O: C, 65.44%; H, 4.97%; N, 25.44%. found: C, 65.38%; H, 4.95%; N, 25.37%.

**Compound (R<sub>8</sub>):** Yield: 89%; m.p. >300<sup>0</sup>C (dec.); IR (KBr,cm<sup>-1</sup>) 3120-3178 cm<sup>-1</sup> (-NH-), 1489-1572 cm<sup>-1</sup> (-N=N-), 3032-3057 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group, 748 cm<sup>-1</sup> (-C-Cl-); <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85 (s, 1H, -CH), 7.43-8.91 (m, 9H, Ar-H), 13.70 (s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>14</sub>ClN<sub>7</sub>O<sub>4</sub>S: C, 65.44%; H, 4.97%; N, 25.44%. found: C, 65.32%; H, 4.85%; N, 25.39%.

**Compound (R<sub>9</sub>):** Yield: 79%; m.p. >300<sup>0</sup>C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120-3178 cm<sup>-1</sup> (-NH-), 1479-1570 cm<sup>-1</sup> (-N=N-), 3032-3057 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2873-2920 cm<sup>-1</sup> (-C-H) stretching of methyl group, 750 cm<sup>-1</sup> (-C-Cl-); <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85 (s, 1H, -CH), 7.43-8.32 (m, 4H, Ar-H), 13.51(s, 1H, -NH); Anal. Calcd. for C<sub>20</sub>H<sub>13</sub>Cl<sub>2</sub>N<sub>7</sub>O: C, 54.81%; H, 2.99%; N, 22.37%. found: C, 54.79%; H, 3.00%; N, 22.30%.

**Compound (R<sub>10</sub>):** Yield: 87%; m.p. >300<sup>0</sup>C (dec.); IR (KBr,cm<sup>-1</sup>) : 3120-3178 cm<sup>-1</sup> (-NH-), 1543-1572 cm<sup>-1</sup> (-N=N-), 3032-3057 cm<sup>-1</sup> (-C-H) stretching of aromatic rings, 2810-2955 cm<sup>-1</sup> (-C-H) stretching of methyl group; <sup>1</sup>H NMR (400 MHz, DMSO- *d*<sub>6</sub>) δ 2.46 (s, 3H, Me), 2.85 (s, 1H, -CH), 7.43-8.91 (m, 8H, Ar-H), 11.28 (s, 1H, -NH), 13.51(s, 1H, -NH); Anal. Calcd. for C<sub>14</sub>H<sub>11</sub>N<sub>7</sub>O: C, 57.33%; H, 3.78%; N, 33.43%. found: C, 57.31%; H, 3.72%; N, 33.40%.

## RESULTS AND DISCUSSION

The derivatives of 3-methyl-1-phenyl-4-[(E)-2-{1H-pyrazolo [3,4-b] quinolin-3-yl} diazen-1-yl]-4,5-dihydro-1H-pyrazol-5-one were synthesized using following stage. 2-choloroquinolin-3-carbaldehyde (1) has been synthesized using the Vilsmeier-Haack reagent as per the literature [16]. This on further reaction with hydroxyl amine hydrochloride by using sodium acetate as catalyst gave oxime (2) in high yield and purity. Compound (3) was obtained in high yield and purity by condensation of compound (2) and thionyl chloride in benzene. The product (3) was then treated with hydrazine hydrate in ethanol to produce compound (4). Compound (4) on diazotization and coupling with phenyl pyrazolones derivative gave scheme 1.

### Spectral properties

The visible absorption spectroscopic properties of the dyes were recorded in DMF and water. The absorption maxima (λ<sub>max</sub>) of all the dyes falls in the range 422–542 nm in DMF and water,

the values are given in Table 1. Absorption maxima of dyes (D<sub>1</sub>–D<sub>5</sub>) were recorded in water and dyes (D<sub>6</sub>–D<sub>10</sub>) were recorded in DMF, showed in Table 1. As far as absorption maxima are concerned,  $\lambda_{\text{max}}$  values are directly proportional to the electronic power of the substituent in the coupled ring system. The presence of electron donating and electron attracting groups at suitable position in the coupled ring affect the absorption characteristics of the dye.

All the compounds showed a characteristic band at 1450-1590 cm<sup>-1</sup> for the -N=N group. The band at 2810-2995 cm<sup>-1</sup> is due to the C-H stretching of methyl groups. The characteristic band 3120-3178 cm<sup>-1</sup> is attributed to N-H stretching of secondary amine group. The band appears at 3032-3057 cm<sup>-1</sup> corresponding to C-H stretching of aromatic rings. The band at 740-785 cm<sup>-1</sup> is due to a C-Cl stretching. The <sup>1</sup>H-NMR spectra of all the synthesized compounds based on 1H-pyrazolo[3,4-b]quinolin-3-amine shows important signals at their respective positions, confirming the structures.

#### *Dyeing properties of dyes*

All the dyes D<sub>1</sub>–D<sub>5</sub> were applied on silk, wool and nylon fabrics and D<sub>6</sub>–D<sub>10</sub> were applied on polyester fabrics in 2% shade according to the usual procedure [17]. Dyeing was carried out at pH 3.0 on nylon, silk and wool pH was adjusted to 3.0 by dilute acetic acid and formic acid while for D<sub>6</sub>–D<sub>10</sub> pH was adjusted 7.0 by soda ash solution. The variations in the hues of the dyed fabric results from the alteration in the coupling components.

The heterocyclic monoazo dyes were applied at a 2 % depth on nylon, wool silk and polyester fabrics. Their dyeing properties are shown in Tables 2. These dyes gave a wide range of colors varying from light yellow to dark yellow with good levelness, brightness and depth on the fabrics. The variation in the shades of the dye fabric results from both the nature and position of the substituent present on the diazotized compound. The dyeing showed fair to good fastness to light, washing and rubbing fastnesses. A remarkable degree of smoothness after washing was observed. This may be attributed to the good penetration into and affinity of the dye molecule for the structure of the fabrics.

#### *Exhaustion and fixation study*

Dye bath exhaustion (%) and dye bath fixation (%) of dyed fabric were determined according to reported method [18]. The date of exhaustion and fixation are shown in Table 1. The exhaustion of dyes D<sub>1</sub>–D<sub>5</sub> for nylon ranges from 64% to 70%, for wool varied from 64% to 71% and for silk varied from 67% to 78%. The fixation of dyes D<sub>1</sub>–D<sub>5</sub> for nylon varied from 83% to 90%, for wool varied from 82% to 93% and for silk varied from 84% to 93% while the exhaustion of dyes D<sub>6</sub>–D<sub>10</sub> for polyester ranges from 67% to 78% and fixation of dyes D<sub>6</sub>–D<sub>10</sub> for polyester varied from 84% to 93%.

#### *Fastness properties*

Data on fastness properties of light, wash and rubbing are shown in Table 2. The fastness properties of light were assessed in accordance with BS: 1006-1378 [19], the wash fastness test in accordance with IS: 765-1979 [20] and rubbing fastness carried out with cork meter (Atlas) in accordance with AATCC-1961 [21]. The light fastness was moderate to good for nylon, wool, silk and polyester while wash and rubbing fastness was good to excellent for nylon, wool, silk and polyester.

Table 1 Exhaustion and Fixation data of dyes (D<sub>1</sub>-D<sub>10</sub>)

Dye No	Shade of dyed fiber	$\lambda_{\max}$ (nm)	Exhaustion ( % )				Fixation			
			S	W	N	P	S	W	N	P
D <sub>1</sub>	Light Yellow	420	67.35	71.80	67.15	-	92.95	90.75	83.65	-
D <sub>2</sub>	Dark yellow	434	73.45	64.15	68.40	-	88.25	85.12	85.72	-
D <sub>3</sub>	Yellow	428	70.30	68.25	64.22	-	84.00	82.05	90.55	-
D <sub>4</sub>	Yellow	428	68.25	70.45	69.12	-	86.45	88.15	84.85	-
D <sub>5</sub>	Yellow	426	78.00	64.55	70.44	-	85.75	93.90	85.90	-
D <sub>6</sub>	Light Yellow	420	-	-	-	67.35	-	-	-	92.95
D <sub>7</sub>	Light Yellow	420	-	-	-	73.45	-	-	-	88.25
D <sub>8</sub>	Yellow	430	-	-	-	70.30	-	-	-	84.00
D <sub>9</sub>	Yellow	440	-	-	-	68.25	-	-	-	86.45
D <sub>10</sub>	Light Yellow	430	-	-	-	78.00	-	-	-	85.75

*D<sub>1</sub> to D<sub>5</sub>: Acid dyes, D<sub>6</sub>-D<sub>10</sub>: Dispersed dyes*

Table 2 Fastness properties of dyes (D<sub>1</sub>-D<sub>10</sub>)

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

*D<sub>1</sub> to D<sub>5</sub>: Acid dyes, D<sub>6</sub>-D<sub>10</sub>: Dispersed dyes*

*S - Silk, W - Wool, C - Nylon, P - Polyester.*

*Light fastness: (Grading: 1- poor, 2- slight, 3- moderate, 4- fair, 5- good, 6- very good).*

*Wash and rubbing fastness: (Grading: 1- poor, 2- fair, 3- good, 4- very good, 5- excellent).*

## CONCLUSION

Dyeing study reveals that the fastness on acid and dispersed dyes on all fibers is moderate to good. Washing fastness of the dyes for all fibers was found to be good to excellent. Fastnesses to rubbing of dyed patterns were found to be very good to excellent for all dyes on all fibers. These dyes give mostly yellow shades on wool, silk, nylon and polyester fabric having good fastness properties. The variations in the hues of the dyed fabric results from both, the nature and position of the substituent present on the coupler ring. The exhaustion and fixation of these dyes are very good; this indicates that the dyes have good solubility and affinity towards the fabric. The observed remarkable degree of levelness after washing indicates good penetration and affinity of these dyes to the fabric.

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