







Where, R = various 2-((4-aminophenyl)sulfonyl)ethyl hydrogen sulfato cyanurated coupling components to synthesized PK<sub>1</sub> to PK<sub>12</sub> (Table-1)

## 4. Results and Discussion

### Dyeing of fibres

All the dyes were applied on silk, wool and cotton fabrics in 2% shade according to usual procedure [20]. The variation in the hues of the dyed fabric results from both the nature and position of the substituent present on the coupler ring. The remarkable degree of levelness after washing indicates good penetration and affinity of these dyes to the fabric.

A series of reactive dyes were synthesized in order to evaluate their stability for dyeing cotton, wool and silk fabrics. The yield of these dyes ranges from 78 to 88 %. The structures were identified by analytical and spectral evidences.

### IR and <sup>1</sup>H NMR spectral study

IR spectra of all the dyes, in general showed O-H and N-H stretching vibrations in the region 3570-3588 cm<sup>-1</sup>, 3401-3418 cm<sup>-1</sup> respectively, C-H stretching vibration at 3270-3288 cm<sup>-1</sup>, N=N stretching vibration at 1610-1629 cm<sup>-1</sup>, C-N stretching vibration at 1507-1548 cm<sup>-1</sup>, S=O stretching vibration at 1033-1168 cm<sup>-1</sup>, C-Cl stretching vibration at 709-724 cm<sup>-1</sup>, NO<sub>2</sub> stretching vibration at 1521-1529 cm<sup>-1</sup> (Table-2). The <sup>1</sup>H NMR spectra of representative dyes showed all the signals (Table-2).

### Exhaustion and fixation study

The percentage exhaustion of 2% dyeing on silk fabric showed from 65.67% to 78.99%, for wool fabric showed from 65.41% to 76.46% and for cotton fabric showed from 66.02% to 75.98%. The percentage fixation of 2% dyeing on silk fabric showed from 70.39% to 93.78 %, for wool fabric showed from 75.97% to 92.90% and for cotton fabric showed from 73.39% to 95.31 %, (Table-3).

### Spectral properties

*Electronic spectra:* From the data reported in Table-3, it is apparent that the value of  $\lambda_{max}$  depends on the coupling components used. The colour change observed for each dye is due to the oscillation of electrons and the presence of additional substituents.

*Visible Absorption Spectroscopic Properties of Dye:* The visible absorption spectroscopic properties of dyes were recorded in water (Table-3). The colour of the dye is affected by substituents in the coupling constituent.

### Fastness properties

The light fastness was study according to BS: 1006-1978 [21]. The rubbing fastness test was carried out with a Crock meter (Atlas) in accordance with AATCC-1961 [22] and the wash fastness test in accordance with IS: 765-1979 [23]. The light fastness of all the dyes showed moderate to good on silk, wool and cotton. The wash fastness of all the dyes showed good to excellent on silk, wool and cotton and the rubbing fastness (dry and wet) of all the dyes showed good to excellent rubbing fastness on wool, silk and cotton fabrics (Table-4).

## 5. Conclusions

3-(4-(4-Amino-3-nitrobenzyl)-2-nitrophenyl)-2-phenylquinazolin-4(3H)-one was diazotized and coupled with various 2-((4-aminophenyl)sulfonyl)ethyl hydrogen sulfato cyanurated coupling components to give the corresponding monoazo reactive dyes (PK<sub>1</sub> to PK<sub>12</sub>). These dyes gave pink, orange, cream, maroon and yellow shade on wool, silk and cotton and showed very good fastness properties. Exhaustion and fixation of these dyes are very good in order. These dyes PK<sub>1</sub> PK<sub>3</sub> PK<sub>4</sub> PK<sub>5</sub> PK<sub>7</sub> and PK<sub>11</sub> gave better light fastness. The introduction of a triazine group to the dye molecule improves the degree of exhaustion and fixation of the dyes. The remarkable degree of levelness after washing indicates the good penetration and affinity of these dyes to the fabric.

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**Table 1:** Physical properties of dyes PK<sub>1</sub>-PK<sub>12</sub>

Dye No.	R	Molecular Formula	Molecular Weight (g.)	Yield %	M.P °C	$\lambda_{max}$ nm	Nitrogen %	
							Found	Calcd.
PK1	H-acid	C <sub>48</sub> H <sub>31</sub> CIN <sub>11</sub> Na <sub>3</sub> O <sub>18</sub> S <sub>4</sub>	1282	80	260	535	12.11	12.01
PK2	Gamma acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>15</sub> S <sub>3</sub>	1180	81	220	509	13.15	13.05
PK3	J-acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>15</sub> S <sub>3</sub>	1180	80	240	413	13.18	13.05
PK4	N-Methyl J-acid	C <sub>49</sub> H <sub>34</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>15</sub> S <sub>3</sub>	1194	85	280	495	12.97	12.90
PK5	N-Phenyl J-acid	C <sub>54</sub> H <sub>36</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>15</sub> S <sub>3</sub>	1256	84	220	490	12.36	12.26
PK6	S-acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>15</sub> S <sub>3</sub>	1180	80	240	420	13.21	13.05
PK7	Naphthionic acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>14</sub> S <sub>3</sub>	1164	87	205	412	13.32	13.23
PK8	Peri acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>14</sub> S <sub>3</sub>	1164	79	170	410	13.15	13.23
PK9	Bronner acid	C <sub>48</sub> H <sub>32</sub> CIN <sub>11</sub> Na <sub>2</sub> O <sub>14</sub> S <sub>3</sub>	1164	83	140	419	13.28	13.23
PK10	Tobias acid	C <sub>48</sub> H <sub>33</sub> CIN <sub>11</sub> NaO <sub>11</sub> S <sub>2</sub>	1062	79	230	397	14.62	14.50
PK11	K-acid	C <sub>48</sub> H <sub>30</sub> CIN <sub>11</sub> Na <sub>4</sub> O <sub>20</sub> S <sub>5</sub>	1368	88	150	379	11.18	11.26
PK12	Koch acid	C <sub>48</sub> H <sub>30</sub> CIN <sub>11</sub> Na <sub>4</sub> O <sub>20</sub> S <sub>5</sub>	1368	78	170	512	11.40	11.26

Table 2: IR and <sup>1</sup>H NMR data of dyes PK<sub>1</sub>-PK<sub>12</sub>

Dye No.	IR (KBr) cm <sup>-1</sup>
PK <sub>1</sub>	3584 (O-H), 3416 (N-H), 3287 (C-H), 1635 (C=O), 1627 (N=N), 1548 (C-N), 1163, 1057 (S=O, asy & sym), 803 (C-S), 714 (C-Cl), 1524 (NO <sub>2</sub> ).
PK <sub>2</sub>	3586 (O-H), 3407 (N-H), 3279 (C-H), 1626 (C=O), 1624 (N=N), 1539 (C-N), 1153, 1050 (S=O, asy & sym), 803 (C-S), 714 (C-Cl), 1525 (NO <sub>2</sub> ).
PK <sub>3</sub>	3581 (O-H), 3411 (N-H), 3282 (C-H), 1629 (C=O), 1629 (N=N), 1537 (C-N), 1154, 1057 (S=O, asy & sym), 817 (C-S), 713 (C-Cl), 1522 (NO <sub>2</sub> ).
PK <sub>4</sub>	3570 (O-H), 3401 (N-H), 3275 (C-H), 1620 (C=O), 1610 (N=N), 1507 (C-N), 1152, 1052 (S=O, asy & sym), 818 (C-S), 709 (C-Cl), 1529 (NO <sub>2</sub> ).
PK <sub>5</sub>	3578 (O-H), 3408 (N-H), 3270 (C-H), 1621 (C=O), 1624 (N=N), 1533 (C-N), 1166, 1065 (S=O, asy & sym), 807 (C-S), 724 (C-Cl), 1523 (NO <sub>2</sub> ).
PK <sub>6</sub>	3571 (O-H), 3409 (N-H), 3271 (C-H), 1636 (C=O), 1627 (N=N), 1538 (C-N), 1153, 1054 (S=O, asy & sym), 811 (C-S), 720 (C-Cl), 1526 (NO <sub>2</sub> ).
PK <sub>7</sub>	3583 (O-H), 3418 (N-H), 3288 (C-H), 1630 (C=O), 1620 (N=N), 1531 (C-N), 1168, 1060 (S=O, asy & sym), 809 (C-S), 710 (C-Cl), 1522 (NO <sub>2</sub> ).
PK <sub>8</sub>	3580 (O-H), 3406 (N-H), 3270 (C-H), 1634 (C=O), 1627 (N=N), 1530 (C-N), 1154, 1056 (S=O, asy & sym), 811 (C-S), 714 (C-Cl), 1524 (NO <sub>2</sub> ).
PK <sub>9</sub>	3587 (O-H), 3406 (N-H), 3277 (C-H), 1635 (C=O), 1620 (N=N), 1529 (C-N), 1165, 1050 (S=O, asy & sym), 812 (C-S), 716 (C-Cl), 1529 (NO <sub>2</sub> ).
PK <sub>10</sub>	3580 (O-H), 3412 (N-H), 3270 (C-H), 1640 (C=O), 1628 (N=N), 1535 (C-N), 1150, 1058 (S=O, asy & sym), 800 (C-S), 714 (C-Cl), 1525 (NO <sub>2</sub> ).
PK <sub>11</sub>	3573 (O-H), 3409 (N-H), 3272 (C-H), 1637 (C=O), 1626 (N=N), 1539 (C-N), 1165, 1069 (S=O, asy & sym), 808 (C-S), 710 (C-Cl), 1521 (NO <sub>2</sub> ).
PK <sub>12</sub>	3588 (O-H), 3411 (N-H), 3286 (C-H), 1639 (C=O), 1621 (N=N), 1538 (C-N), 1164, 1065 (S=O, asy & sym), 811 (C-S), 724 (C-Cl), 1522 (NO <sub>2</sub> ).

Dye No.	<sup>1</sup> H NMR (DMSO) Chemical shift in δ ppm
PK <sub>1</sub>	3.62-3.64 (t, 2H, -CH <sub>2</sub> -), 3.81 (s, 2H, -CH <sub>2</sub> -), 3.98 (s, 2H, 2, -NH), 4.22 (t, 2H, -CH <sub>2</sub> -), 5.36 (s, 1H, -OH), 6.96-8.67 (m, 22H, Ar-H)

Abbreviations in <sup>1</sup>H NMR data: s, singlet, d, doublet, t, triplet, m, multiplet.

Table 3: Result of Exhaustion and Fixation study of reactive dyes on silk, wool, cotton and Shade on fabrics

Dye No.	Shade on silk fabrics	Shade on wool fabrics	Shade on cotton fabrics	Exhaustion (%)			Fixation (%)		
				S	W	C	S	W	C
PK <sub>1</sub>	Pink	Dark maroon	Pink	73.76	74.53	70.56	85.45	92.08	92.73
PK <sub>2</sub>	Light maroon	Light maroon	Yellowish pink	68.97	71.78	70.90	77.90	90.92	86.84
PK <sub>3</sub>	Light orange	Light Orange	Light orange	72.56	65.64	66.87	92.95	88.51	76.23
PK <sub>4</sub>	Orange	Orange	Orange	73.87	72.28	75.76	93.78	89.06	90.73
PK <sub>5</sub>	Dark orange	Dark orange	Dark orange	68.97	76.46	73.90	74.67	88.40	95.31
PK <sub>6</sub>	Golden	Cream	Cream	65.98	67.75	64.78	72.76	87.89	78.95
PK <sub>7</sub>	Yellow	Dark Yellow	Yellow	74.97	76.97	72.87	92.74	91.24	93.73
PK <sub>8</sub>	Cream	Yellow	Light cream	69.97	69.99	67.97	75.98	75.98	74.44
PK <sub>9</sub>	Yellow	Pale yellow	Pale yellow	76.98	65.41	66.02	91.54	77.77	76.87
PK <sub>10</sub>	Dark yellow	Dark orange	Dark cream	78.99	70.54	75.98	93.63	92.90	75.89
PK <sub>11</sub>	Yellow	Light Yellow	Light cream	78.97	68.62	71.09	87.52	90.56	73.39
PK <sub>12</sub>	Dark cream	Dark yellow	Pale yellow	65.67	77.77	69.87	70.39	78.54	94.87

Determined in water at 28 °C at 2 × 10<sup>-3</sup> M dye concentrations. S = Silk, W = Wool, C = Cotton.

Table 4: Fastness properties of dyes PK<sub>1</sub>-PK<sub>12</sub>

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

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

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