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# Bi-functional Cold Brand Reactive Dyes with Urea as a Bridge Group: Synthesis, Characterization and Dyeing Performance on Various Fibers

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**ABSTRACT**

Bi-functional reactive dyes from the Cold brand that have exceptional dyeing capabilities. The intermediate for bi-functional cold-brand reactive dyes is 3, 3'-Disulpho-4,4'-diamino diphenyl urea, which is synthesized through the interaction of one mole of urea aqueous solution with two moles of 1,4-diaminobenzene-2-sulphonic acid. In this bi-functional reactive dye system, urea was utilized as a bridge molecule to give the reactive dyes significant water solubility. Incorporating various cyanurated coupling components, different colors are produced. It has good dyeing properties and provides a wide spectrum of shades on various fibres.

**Keyword:** Cold brand, Reactive dyes, Bi-functional, Exhaustion, Fixation, Dyeing properties.

## INTRODUCTION

Reactive dye focuses mostly on the chemical group and chromophore group in the domain of colour. By introducing a new chemical group that improves the dyes dyeing capabilities as well as their shades[1]. The colors, solubility, and fastness properties of dyes can be improved by altering the chemical group of the molecules. Reactive dyes have exceptional fastness qualities because of their chemical attachment, pH, reaction time, dyeing procedure, synthetic auxiliary, dyeing temperature, and dye category[2]. Reactive dyes that are bi-functional provide a wide range of colour spectrum and are highly stable at high temperatures. Organic compound dye molecules that absorb light and produce a variety of colors[3,4]. Dyes compounds with chromophore (visible spectrum-absorbing) and reflect (visible spectrum-reflecting) properties[5]. Dyeing is done to give things like skin, clothing, painting and printing paper colour. They are capable of repairing with cellulose fibre[6]. It has two amine and cyanurated systems that formed covalent bonds with fibers. This bi-functional cold brand reactive dyes presence of multiple functional group and like Sulphonic (-$SO_3H$ gr), Hydroxyl

(-OH gr), Amine (-$NH_2$ gr), and Carbonyl (-C=O gr), they are act as Auxochromes which intensify the colour of Synthesized dyes. When urea is used in the production, the dye molecules become very soluble[7]. Therefore, the goal of the synthesis is to create reactive dyes with good dyeing properties and greater solubility due to urea bridging[8,9].

## MATERIAL AND METHODS

During the synthesis of these reactive dyes, reaction is monitored by the TLC method, IR spectra recorded on the Bruker FTIR Spectrophotometer and NMR Spectroscopy is on Jeol delta NMR using TMS as standard.

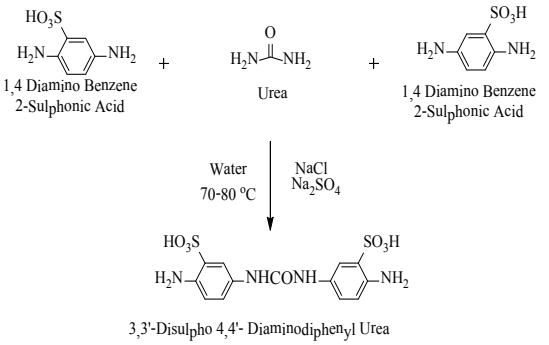
**Synthesis of dyes is carried in four stage, which shown below[10,11]**

**Step: 1**. Synthesis of 3, 3'-Disulpho 4, 4'diamino diphenyl urea is been carried by the reaction between the 1, 4-Diamino benzene 2-sulphonic acid and aqueous boiled solution of urea. This reaction is carried out at 70-80°C for 72 hours. During the reaction, aqueous solution of sodium sulphate is added. Then sodium chloride added after the completion of the reaction and wash with the hot water. Then dried it at 80°C for 5 hours. its purification of it will be done using acetone. Reflux it for 1 hours. at 50-55°C. Cool it and filter it. %yield: 75-80%.

**Step: 2**. In this stage, 3, 3'-Disulpho 4, 4'diamino diphenyl urea is diazotized at 0-5°C. 50% solution of hydrochloric acid and aqueous solution of sodium nitrite. Stir the reaction for 1 hours at 0-5°C. And filter and wash with acetone. %yield: 72-75%.

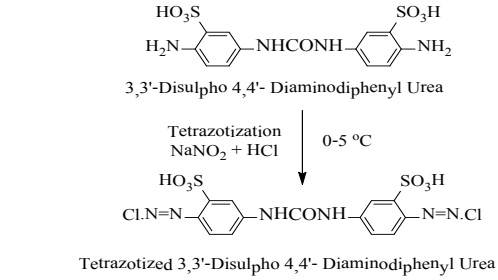
**Step: 3.** Cynuration of H-acid, reaction between Cyanuric chloride and H-acid. Firstly, dissolved the H-acid in solution of sodium carbonate and Cyanuric chloride in acetone, then added them at 0-5°C, till get clear solution of it. Then solution is used to for coupling with diazotized moiety.

**Step: 4.** In this stage Diazotized solution and Cynurated solution of acid is coupled at 0-5°C. Stir it for 1 hours and filter it and wash with acetone. Dry it at 55°C. For 1 hours %yield: 75-85%.

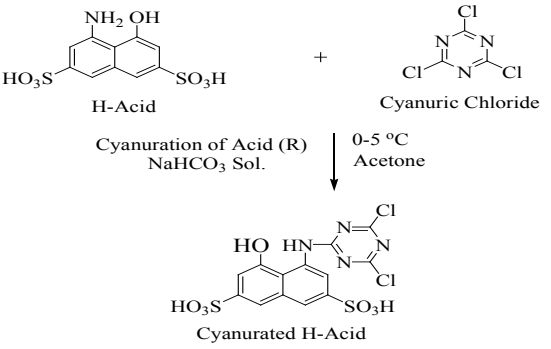



**Step:- 01 Syanthesis of 3,3'-Disulpho 4,4'- Diaminodiphenyl Urea**

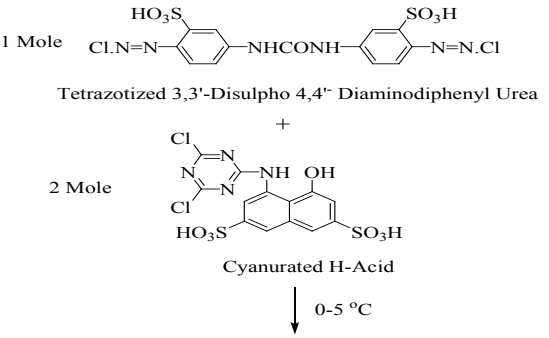
Step:- 02 Tetrazotization of 3,3'-Disulpho 4,4'- Diaminodiphenyl Urea




**Step:- 02 Tetrazotization of 3,3'-Disulpho 4,4'- Diaminodiphenyl Urea**




**Step:- 03 Cyanuration of H-Acid(R)**




**Step:- 04 Coupling of Tetrazotized 3,3'-Disulpho 4,4'-Diaminodiphenyl Urea with Cyanurated H-Acid**

Where Marked Box which indicate Different Coupling Compound (R).

**Table 1: Synthesized Bi-functional cold brand reactive dyes using Different Coupler**

| Dye No | Coupling Component (R) | Molecular Formula | M. Wt. g/mole | %yield |
|---|---|---|---|---|
| R-01 | H-acid | $C_{39}H_{24}Cl_4N_{14}O_2S_6$ | 1355.83 | 80% |
| R-02 | K-acid | $C_{39}H_{24}Cl_4N_{14}O_2S_6$ | 1355.86 | 82% |
| R-03 | J-acid | $C_{39}H_{24}Cl_4N_{14}O_{15}S_4$ | 1195.92 | 85% |
| R-04 | N-Methyl J-acid | $C_{41}H_{28}Cl_4N_{14}O_{15}S_4$ | 1223.95 | 80% |
| R-05 | N-phenyl J-acid | $C_{51}H_{32}Cl_4N_{14}O_{15}S_4$ | 1347.98 | 75% |
| R-06 | Peri acid | $C_{39}H_{24}Cl_4N_{14}O_{13}S_4$ | 1163.93 | 78% |
| R-07 | Tobias acid | $C_{39}H_{24}Cl_4N_{14}O_{13}S_4$ | 1163.93 | 80% |
| R-08 | Sulpho Tobias acid | $C_{39}H_{24}Cl_4N_{14}O_{19}S_6$ | 1323.84 | 75% |
| R-09 | Laurent acid | $C_{39}H_{24}Cl_4N_{14}O_{13}S_4$ | 1163.93 | 79% |
| R-10 | Gamma acid | $C_{39}H_{24}Cl_4N_{14}O_{15}S_4$ | 1195.92 | 80% |
| R-11 | Bronner's acid | $C_{39}H_{24}Cl_4N_{14}O_{13}S_4$ | 1163.93 | 82% |
| R-12 | N-Methyl Gamma acid | $C_{41}H_{28}Cl_4N_{14}O_{15}S_4$ | 1223.95 | 85% |

**Spectral Analysis[12,13,14,15]**

Spectral date of the synthesized cold brand reactive dyes is being studies using IR Spectroscopy, $^1$H NMR and $^{13}$C-NMR spectroscopy.

**Structure of R-02.**

**Structure of R-10.**

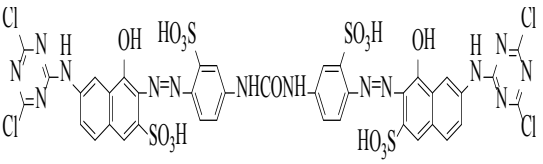


**Table: 2 IR, NMR (1H & C-13) Spectra of Bi-Functional Cold Brand reactive dyes.**

| Dye No | IR Spectra (ATR mode) | $^1$H NMR& $^{13}$C-NMR (DMSO $d_6$) |
|---|---|---|
| R-02 | -$SO_3$H gr. (1207.84 cm$^{-1}$), -OH gr. (3432.81 cm$^{-1}$), -NH gr. (2990.37 cm$^{-1}$), -Cl gr. (676.77 cm$^{-1}$), -CN gr. (1654.28 cm$^{-1}$), -C=O gr. (1732.352 cm$^{-1}$). | $^1$H NMR:7.675-8.841 (d, J-9.2 Hz 12 H, Ar. H), 2.456-2.470 (S, 4 H, -NH gr.), 2.039 (d, J-3.1 Hz, 6H, -SO3H gr.), 9.414-9.494 (d, J-32 Hz, 2H, -OH gr.) $^{13}$C-NMR:115.4 (2C, s), 116.6 (2C, s), 117.7-117.8 (4C, 117.7 (s), 117.7 (s)), 118.1 (2C, s), 122.5 (2C, s), 124.6 (2C, s), 129.1 (2C, s), 132.1 (2C, s), 134.3-134.4 (4C, 134.3 (s), 134.3 (s)), 138.7 (2C, s), 139.0 (2C, s), 139.5 (2C, s), 145.3 (2C, s), 153.0 (1C, s), 155.6 (2C, s), 160.0 (2C, s), 169.3 (4C, s). |
| R-10 | -$SO_3$H gr. (1255.35 cm$^{-1}$) -OH gr. (3392.35 cm$^{-1}$) -NH gr. (3109.40 cm$^{-1}$) -Cl gr. (696.30 cm$^{-1}$) -CN gr. (1653.99 cm$^{-1}$) -C=O gr. (1791.87 cm$^{-1}$) | $^1$H NMR:5.441-7.803 (d, J-6.9 Hz, 12 H, Ar. H), 3.256-3.559 (d, J-15.3 Hz, 4H, -NH gr.), 0.866-2.460 (d, J-33.6, 4H, -SO3H gr.), 8.364 (S, 2H, -OH gr.) $^{13}$C-NMR:113.7 (2C, s), 115.4 (2C, s), 116.6 (2C, s), 117.7 (2C, s), 120.0 (2C, s), 122.5 (2C, s), 125.1 (2C, s), 128.9 (2C, s), 132.1 (2C, s), 134.3-134.4 (4C, 134.3 (s), 134.3 (s)), 138.6-138.8 (4C, 138.7 (s), 138.7 (s)), 139.5 (2C, s), 145.3 (2C, s), 153.0 (1C, s), 155.6 (2C, s), 160.0 (2C, s), 169.3 (4C, s). |

**Dyeing Study of Synthesized dyes(R- 1 to 12)[16,17]**

At the Sir P. T. Sarvajanik College of Science, Surat, India, the dyeing study of the synthetic dyes was carried out. Studies on the use of synthetic dyes on cotton, silk, and wool fabrics.

**Study of % Exhaustion and % Fixation of Cotton Fiber**

Dye (40 g) is dissolved in the cold water (80 mL) to get clear solution of it, and before dyeing cotton fabric is rinse with the cold water. Dyeing solution is making 100 mL with cold water. Take 20 mL of dyeing solution, 2 mL of 10% W/V sodium carbonate solution, and 20 mL of cold water stir it well for 15 min and maintain at 30°C. Enter cotton fiber (2.0 g) in the dyeing solution. Raise the temperature and maintain it for 30 min at 80°C. And at this temperature at Glauber salt solution 2 mL (40% W/V) to achieve a good exhaustion and maintain further 30 minute. And the soaping and drying g of the dyed cotton fabrics.

Substrate of dyeing: 2.0 g cotton fiber.

Taken Dye Amount: 40 g Fixation study dye pattern: 0.1 g cotton.

Method of spectral Study: Water for Exhaustion and Sulphuric acid for Fixation.

**Table 3: %Exhaustion and %Fixation Study of Synthesized bi-functional reactive dyes on Cotton Fibre**

| Dye No | Amount of Remained in dye bath X mg | Amount of dye exhausted from dye bath (40-X=Ymg) | %Exhaustion= Ymg×100/40 (mg) | Amount of Dye in 0.1 g of dyeing (a mg) | Amount of dye in 2.0 g of dyeing (total wt. 20 a=Zmg) | % Fixation= Z mg×100/Y(mg) |
|---|---|---|---|---|---|---|
| R-01 | 4.2 | 35.8 | 89.5 | 1.21 | 24.2 | 67.59 |
| R-02 | 6.4 | 33.6 | 84 | 1.18 | 23.6 | 70.23 |
| R-03 | 3.1 | 36.9 | 92.25 | 1.15 | 23.0 | 62.33 |
| R-04 | 5.4 | 34.6 | 86.5 | 1.08 | 21.66 | 62.42 |
| R-05 | 5.0 | 35.0 | 87.5 | 1.11 | 22.20 | 63.42 |
| R-06 | 6.2 | 33.8 | 84.5 | 1.15 | 23.00 | 68.04 |
| R-07 | 8.3 | 31.7 | 79.25 | 1.17 | 23.4 | 73.81 |
| R-08 | 8.6 | 31.4 | 78.5 | 1.10 | 22.00 | 70.06 |
| R-09 | 7.2 | 32.8 | 82.0 | 1.13 | 22.60 | 68.90 |
| R-10 | 6.3 | 33.7 | 84.25 | 1.16 | 23.20 | 68.84 |
| R-11 | 7.8 | 32.2 | 80.5 | 1.17 | 23.40 | 72.67 |
| R-12 | 5.1 | 34.9 | 87.25 | 1.12 | 22.4 | 64.18 |

**Study of % Exhaustion and % Fixation of Silk Fiber**

Dye (40 mg) is dissolved in the cold water (80 mL) to get clear solution of it, and before dyeing silk fabric is rinse with the cold water. Dyeing solution is make 100 mL with cold water. Take 20 mL of dyeing solution, 2 mL of 10% W/V Acetic acid solution, and 20 mL of cold water stir it well for 15 min and maintain at 30°C. Enter silk fiber (2.0 g) in the dyeing solution. Raise the temperature and maintain it for 30 min at 80°C. And at this temperature at Glauber salt solution 2 mL (40% W/V) to achieve a good exhaustion and maintain further 30 minute. And the soaping and drying g of the dyed silk fabrics.

Substrate of dyeing: 2.0 g silk fiber.
Taken Dye Amount: 40 mg.
Fixation study dye pattern: 0.1 g silk.
Method of spectral Study: Water for Exhaustion and Sulphuric acid for Fixation.

**Table 4: %Exhaustion and %Fixation Study of Synthesized bi-functional reactive dyes on silk Fibre**

| Dye No | Dye remained in dye bath X mg | Dye exhausted from dye bath (40- X=Y mg) | %Exhaustion Y mg×100/40 (mg) | Dye in 0.1 g of dyeing (a mg) | Dye in 2.0 g of dyeing (total wt. 20 a=Z mg) | %Fixation=Z mg ×100/Y (mg) |
|---|---|---|---|---|---|---|
| R-01 | 1.1 | 38.9 | 97.25 | 1.12 | 22.40 | 57.58 |
| R-02 | 2.3 | 37.7 | 94.25 | 1.20 | 24.00 | 63.66 |
| R-03 | 2.6 | 37.4 | 93.5 | 1.14 | 22.80 | 60.69 |
| R-04 | 2.2 | 37.8 | 94.5 | 1.10 | 22.00 | 58.20 |
| R-05 | 1.5 | 38.5 | 96.25 | 1.12 | 22.40 | 58.18 |
| R-06 | 3.2 | 36.8 | 92.0 | 1.08 | 21.6 | 58.69 |
| R-07 | 3.1 | 36.9 | 92.25 | 1.21 | 24.2 | 65.58 |
| R-08 | 3.5 | 36.5 | 91.25 | 1.10 | 22.00 | 60.27 |
| R-09 | 3.0 | 37 | 92.5 | 1.11 | 22.20 | 60.00 |
| R-10 | 2.8 | 37.2 | 93.0 | 1.10 | 22.00 | 59.13 |
| R-11 | 3.3 | 36.7 | 91.75 | 1.12 | 22.40 | 61.03 |
| R-12 | 1.8 | 38.2 | 95.5 | 1.10 | 22.00 | 57.59 |

**Study of % Exhaustion and % Fixation of wool fiber**

Dye (40 mg) is dissolved in the cold water (80 mL) to get clear solution of it, and before dyeing wool fabric is rinse with the cold water. Dyeing solution is make 100 mL with cold water. Take 20 mL of dyeing solution, 2 mL of 10% W/V formic acid solution, and 20 mL of cold water stir it well for 15 min and maintain at 30°C. Enter wool fiber (2.0 g) in the dyeing solution. Raise the temperature and maintain it for 30 min at 80°C. And at this temperature at Glauber salt solution 2 mL (40% W/V) to achieve a good exhaustion and maintain further 30 minute. And the soaping and drying of the dyed wool fabrics.

Substrate of dyeing: 2.0 g wool fiber.
Taken Dye Amount: 40 mg.
Fixation study dye pattern: 0.1 g wool.
Method of spectral Study: Water for Exhaustion and Sulphuric acid for Fixation.

**Table 5: %Exhaustion and %Fixation Study of Synthesized bi-functional reactive dyes on wool Fibre**

| Dye No | Dye remained in dye bath X mg | Dye exhausted from dye bath(40- X=Y mg) | % Exhaustion= Y mg×100/40 (mg) | Dye in 0.1 g of dyeing (a mg) | Dye in 2.0 g of dyeing (total wt. 20 a=Z mg) | % Fixation= Z mg×100/Y (mg) |
|---|---|---|---|---|---|---|
| R-01 | 2.5 | 37.5 | 93.75 | 1.10 | 22.00 | 58.66 |
| R-02 | 3.6 | 36.4 | 91.0 | 1.12 | 22.40 | 61.53 |
| R-03 | 2.8 | 37.2 | 93.0 | 1.10 | 22.00 | 59.13 |
| R-04 | 5.2 | 34.8 | 87.0 | 1.11 | 22.20 | 63.79 |
| R-05 | 3.4 | 36.6 | 91.5 | 1.13 | 22.60 | 61.74 |
| R-06 | 3.7 | 36.3 | 90.75 | 1.10 | 22.00 | 60.60 |
| R-07 | 3.5 | 36.5 | 91.25 | 1.12 | 22.40 | 61.36 |
| R-08 | 3.1 | 36.9 | 92.25 | 1.11 | 22.20 | 60.16 |
| R-09 | 3.3 | 36.7 | 91.75 | 1.13 | 22.60 | 61.58 |
| R-10 | 2.1 | 37.9 | 94.75 | 1.10 | 22.00 | 58.04 |
| R-11 | 2.7 | 37.3 | 93.25 | 1.10 | 22.00 | 58.98 |
| 1R-12 | 1.8 | 38.2 | 95.5 | 1.12 | 22.40 | 58.63 |

**Table 6: Study of Fastness Property Synthesized bi-functional cold brand reactive dyes (R-1 to 12)**

| Dye No | Shades on Fibers | | | Light fastness | Washing Fastness | Rubbing Fastness |
|---|---|---|---|---|---|---|
| | Cotton | Silk | Wool | | | |
| R-01 | Purple | Purple | Purple | 6 | 5 | 5 |
| R-02 | Cream | Golden | Brown | 2-3 | 2 | 2-3 |
| R-03 | Orange | Orange | Orange | 4-5 | 4-5 | 5 |
| R-04 | Light orange | Brown | Brown | 3-4 | 4-5 | 4-5 |
| R-05 | Brown | Dark brown | Dark brown | 5-6 | 6 | 6 |
| R-06 | Light brown | Brown | Brown | 3-4 | 4-5 | 4-5 |
| R-07 | Light yellow | Golden | Golden | 2-3 | 4-5 | 4-5 |
| R-08 | Cream | Golden | Golden | 1-2 | 2-3 | 2-3 |
| R-09 | Cream | Golden | Golden | 2-3 | 3 | 3 |
| R-10 | Brown | Orange | Orange | 3 | 4-5 | 4-5 |
| R-11 | Cream | Golden | Golden | 2-3 | 4 | 5 |
| R-12 | brown | brown | Brown | 3 | 3 | 3 |

**Abbreviation**

Light Fastness: 1-(poor), 2-(slight), 3-(moderate), 4-(fair), 5-(good), 6-(very good), 7-(excellent)

Washing Fastness: 1-(poor), 2-(slight), 3-(moderate), 4-(fair), 5-(good), 6-(very good), 7-(excellent)

Rubbing Fastness: 1-(poor), 2-(slight), 3-(moderate), 4-(fair), 5-(good), 6-(very good), 7-(excellent)

## RESULT AND DISCUSSION[18,19]

Synthesis of this type of bi-functional reactive dyes have low material cost and having low environmental effect due to its high solubility and higher affinity towards the fibre and minimize the wastage of material[17]. This bi-functional reactive dyes has good exhaustion and fixation properties[18]. In the synthesis of bifunctional cold brand reactive dyes whereas urea as a bridging group, the presence of urea (-NH gr.) is Confirmed by the IR Spectra and also by the NMR spectroscopy.

Dye-02: In the synthesis of Dye-02, K-acid used as a coupling component, whereas presence of functional group, is $–SO_3H$ gr. Stretching at 1207.84 $cm^{-1}$, -OH gr. Stretching at 3432.81 $cm^{-1}$, -NH gr. Stretching secondary amine at 2990.37 $cm^{-1}$, -Cl gr. Stretching 676.77 $cm^{-1}$, -C-N gr. Stretching 1654.28 $cm^{-1}$, and –C=O gr. Stretching at 1732.35 $cm^{-1}$.

And Aromatic Proton (Ar. H-12 H, 7.675-8.841), Secondary Amine Proton (-NH, 4 H, 2.456-2.470), Sulphonic group Proton is at $-SO_3H$ (6 H, 2.039), and alcoholic proton is at -OH (2 H, 9.414-9.494).

Dye-10: In the synthesis of Dye-02, Gamma acid used as a coupling component, whereas presence of functional group, is $–SO_3H$ gr. Stretching at 1255.35

$cm^{-1}$, -OH gr. Stretching at 3392.35 $cm^{-1}$, -NH gr. Stretching secondary amine at 3109.40 $cm^{-1}$, -Cl gr. Stretching 696.30 $cm^{-1}$, -C-N gr. Stretching 1653.99 $cm^{-1}$, and –C=O gr. Stretching at 1791.84 $cm^{-1}$.

And Aromatic Proton (Ar. H-12 H, 5.447-7.803), Secondary Amine Proton (-NH, 4 H, 3.256-3.559), Sulphonic group Proton is at -$SO_3$H (4H, 0.866-2.460), and alcoholic proton is at -OH (2H, 8.364).

From the all this synthesis of cold brand reactive dyes it show significant result about the dyeing application and Exhaustion and fixation. Use of different coupling component it shows excellent results.

For cotton fabric, coupling component K-acid, Tobias Acid, Sulpho Tobias acid, Bronner's acid show good %Exhaustion and %fixation. Give slight to very good rubbing, washing, and light fastness.

For wool fabric, coupling component K-acid, J-acid, Tobias acid, Bronner's Acid show good% Exhaustion and %fixation. Give moderate to good rubbing, washing ad light fastness.

For Silk fabric, coupling Component K-acid, N-methyl J-acid, N-phenyl J-acid, Tobias acid, Laurent acid shows good %Exhaustion and %fixation. Give slight to very good rubbing, washing, light Fastness.

## CONCLUSION

Studying the cold brand bi functional reactive dyes' dyeing capabilities reveals good Properties of exhaustion and fixation. Some of the synthetic dyes have very good capabilities for light fastness, washing fastness, and rubbing fastness. On cotton exhaustion is 92.25 to 78.50%, fixation 73.81 to 62.33%, on silk exhaustion is 97.25 to 91.25% and fixation is 65.58 to59.13% and on wool exhaustion is 93.75 to 87.0% and fixation is 63.79 to 58.04%. And prepare dyes shows the slight to very good rubbing, washing and light fastness to the fibers like cotton, wool, silk.

## ACKNOWLEDGEMENT

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### Conflict of interest

The author declare that we have no conflict of interest.

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