

Synthesis and characterization of novel resorcinol based trisazo reactive dye ligand and its different metal complexes for cotton dyeing

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MUREED ABBAS
SHUMAILA KIRAN
GHULAM HUSSAIN

TAHSIN GULZAR
MIRZA NADEEM AHMAD

ABSTRACT – REZUMAT

Synthesis and characterization of novel resorcinol based trisazo reactive dye ligand and its different metal complexes for cotton dyeing

The goal of this study was to create a new trisazo reactive dye ligand from 6-nitro-2-aminophenol-4-sulphonic acid, resorcinol derivative, and vinyl sulphone para ester, as well as their metal complexes with Fe (II), Ni (II), Cu (II), and Zn (II). The diazotization of 6-nitro-2-aminophenol-4-sulphonic acid was followed by coupling to resorcinol in an alkaline medium for the synthesis of a mono-azo dye ligand. To make reactive dye, diazotized vinyl sulphone para ester was coupled with 6-nitro-2-aminophenol-4-sulphonic acid resorcinol derivative in an alkaline medium. Metal complexes of reactive dyes with 3d transition metals such as iron, nickel, copper, and zinc have also been created. The novel synthesized reactive dyes were applied to cotton to see how well they dyed. Spectro-analytical techniques were used to authenticate the compositions of all newly synthesized substances. The prepared reactive dyes were also used on textiles for dyeing features such as light fastness and wash fastness and were found to have high values of 4–5 on the grey scales, and 4–5 on the blue scales.

Keywords: resorcinol, diazotization, azo dye, coupling reaction, metal complexes, dyeing, fastness properties

Sinteza și caracterizarea noului ligand de colorant reactiv trisazo pe bază de rezorcinol și diferiții săi complecși metalici pentru vopsirea bumbacului

Scopul acestui studiu a fost acela de a crea un nou ligand de colorant reactiv trisazo din acid 6-nitro-2-aminofenol-4-sulfonic, derivat de rezorcinol și vinil sulfonă para ester, precum și complecșii lor metalici cu Fe (II), Ni (II), Cu (II) și Zn (II). Diazotarea acidului 6-nitro-2-aminofenol-4-sulfonic a fost urmată de cuplarea la rezorcinol într-un mediu alcalin pentru sinteza ligandului de colorant mono-azoic. Pentru a crea colorantul reactiv, vinil sulfona para ester diazotat a fost cuplat cu derivatul de rezorcinol al acidului 6-nitro-2-aminofenol-4-sulfonic într-un mediu alcalin. Au fost creați, de asemenea, complecși metalici de coloranți reactivi cu metale de tranziție 3d, cum ar fi fierul, nichelul, cuprul și zincul. Noii coloranți reactivi sintetizați au fost aplicați pe bumbac pentru a se observa procesul de vopsire. Au fost folosite tehnici spectro-analitice pentru identificarea compozițiilor tuturor substanțelor nou sintetizate. Coloranții reactivi preparați au fost utilizați și pe materiale textile pentru determinarea caracteristicilor de vopsire, precum rezistența la lumină și rezistența culorii la spălare și s-au dovedit a avea valori ridicate de 4–5 pe scara de gri și, respectiv, 4–5 pe scara de albastru.

Cuvinte-cheie: rezorcinol, diazotare, colorant azoic, reacția de cuplare, complecși metalici, vopsire, proprietăți de rezistență a culorii

INTRODUCTION

Azo dyes are the most common type of artificial colours, accounting for sixty to seventy per cent of all synthetic dyes now in use [1]. Because of their significantly maximum absorbance values, medium to excellent fastness, good solubility, high technological applications, variety in structures, availability of a wide range of raw ingredients, and simple synthetic techniques, azo dyes are extensively used and synthesized [2]. Azo substances are nitrogen compounds that do not exist in nature and are constantly being studied in technology and academia, azo synthetic dyes are perhaps the most used colourants in the world and make up the biggest class of azo

substances [3]. Numerous azo variants of resorcinol have been studied for a multiple-use because of their suitability for textile fabric [4]. Azo colourants are used in the colouration of fibres, photo electronics, photonic storage devices, biochemical processes printmaking technologies as well as diagnostic, culinary research and biochemistry [5]. In electrical toners, transition metal chelates of azo dye ligands are used as charge regulating materials, dyes in electricity paints, and the electromagnetic separator. Reactive dyes, direct dyes, vat dyes, and metal complex dyes are among the different types of textile dyes. Reactive dyes are frequently used to colour viscose textiles, direct and vat dyes were once used

to colour celluloid materials, but their value has decreased since the advent of reactive dyes [6]. Reactive dyes beat direct dyes in terms of reliability to processing, beautiful hues throughout a diverse variety, high smoothing performance, excellent wash fastness, and improved light fastness [7]. Reactive dyes are the cheapest to manufacture and simplest to use than other dyes. Reactive dyes are chemicals that can combine directly with a surface to create a covalent dye-substrates bonding [8]. Reactive dyes are by far the most frequently utilized category of dyes for colouring cellulosic fabrics due to their great characteristics like dissolution rate, ease of installation, diversity of processing techniques, accessibility of a variety of hues, luminance of vibrant colours, accordance with the desired rinse and light fastness, and reasonable cost [9]. The textile material is dyed using reactive colours in a somewhat alkaline solution [10]. In current studies, six novel trisazo reactive dye and metal complexes were synthesized, characterized, and treated with a cotton cloth.

Simultaneously, spectro-analytical techniques were used to investigate the structure, and qualitative properties of ligand reactive colourant, and its complexes with transition metals.

MATERIALS AND METHODS

Materials

Sigma Aldrich supplied all of the types of products. Standard techniques were used to purify the solvents. The Nicolet IR 100 single beam FTIR spectrometer (Fourier-Transform) was used to generate the FTIR spectra. UV-visible spectrophotometer (UV Genesys spectrophotometer) was used to elucidate the UV-Visible absorption spectrum of all dyestuff's solutions.

Diazotization of 6-napsa and vinyl sulphone para ester

300 ml water was taken and 23.4 g of 6-nitro-2-aminophenol-4-sulphonic acid (6-napsa) was added to it with agitation. 7.3 g NaNO_2 (0.105 moles) was added to it and agitated for 5–10 minutes. Then CuSO_4 (4 g) dissolved in 5 ml of warm water was poured into it instantly and stirred for one hour. In the end, 4–5 g sulfamic acid was introduced to kill extra nitrous acid. Diazotized 6-napsa was prepared. In an ice jacketed beaker, 0.04 moles of vinyl sulphone para ester (11.72 g) were taken. 60 g ice, 120 ml water and 0.12 moles HCl (14.6 ml) were mixed. 0.043 moles of NaNO_2 (2.92 g) were added to it and agitated. The temperature was maintained near 0–5°C by ice addition, and stirrings were sustained for one hour. At the completion of the reaction, 5 g of sulfamic acid was introduced to abolish spare nitrous acid.

Resorcinol solution and coupling with diazotized 6-napsa

0.1 moles of resorcinol (11.1 g) were added in 300 ml water in an ice-jacketed beaker, agitated well. Using a burette, diazotized 6-napsa was mixed dropwise in

resorcinol solution. To retain pH 8.0–8.5, Na_2CO_3 (25%) was added. The beaker was jacked out after the whole addition of diazotized 6-napsa substance, the beaker was jacketed out, and agitation was continued till ambient temperature (25°C). Five equivalent shares of coupled substance were made. 0.02 moles of 6-napsa-resorcinol monoazo dye ligand were present in one part.

Azoreactive anchor attachment

Na_2CO_3 (25%) was used to increase the pH up to 7–7.5 of un-metalized or metalized dye ligand, jacketed with ice, 30 g ice mixed in it, diazoanion compound of vinyl sulfone paraester was added slowly, pH 8–8.5 retained by Na_2CO_3 solution (25%) and the 0–2°C temperature was maintained by ice addition till completion of coupling, agitation was sustained for one hour. Ultimate dye jacketed out, mixed and agitated till room temperature [1].

Metallization

10% HCl solution was applied to decrease pH up to 5.5–6 of un-metalized dye and heated up to 50–60°C. 1–2 drops of conc. H_2SO_4 was added in 0.02 moles of each metal salt in 20 ml water and heated up to 50–60°C to acquire a clear solution. In monoazo dye ligand, respective metal salt solution was added slowly and maintained the temperature at 50–60°C for 30–40 min. Identification of metallization progress was identified by comparative paper chromatography [1].

Dye separation from water by filtration, drying and grinding

The pH was reduced up to 6.8 of synthesized un-metallized 6-napsa-resorcinol trisazo reactive dye or 6-napsa-resorcinol metal chelates trisazo reactive dye by conc. HCl. By salting out on a volume basis, separation was done. For about three hours, the whole dye was stirred for good filtration. Filtration of dye was carried out by using an aspirator; an oven was used at 50–60°C to dry the synthesized trisazo reactive dye (residue) and was saved for further applications and characterization after complete drying and grinding of dye.

Dyeing procedure

Mercerized cotton cloths were dyed with reactive dyes and prepared for the current research. The "Tumble Dyeing" procedure was utilized for colouring cotton cloth. The lab scale dyeing recipe was used as, dyeing salt NaCl (5.0 g) and Na_2CO_3 (0.5 g), one drop of 5 M NaOH, X gram weight of cotton textile. One per cent solution of reactive dye was taken and dyed at a rate of six per cent, three per cent and one per cent concerning X (weight of cotton cloth). 9 g of one per cent dye solution was added in 91 g of water for six per cent dyeing of cotton cloth, 4.5 g of one per cent dye was added in 95.5 g of water for three per cent dyeing of cotton fabric, and 1.5 g of one per cent dye solution added in 98.5 g water for one per cent dyeing of cotton cloth, when X was 1.5 g. 45 minutes at 85°C was the dyeing duration (figure 1).

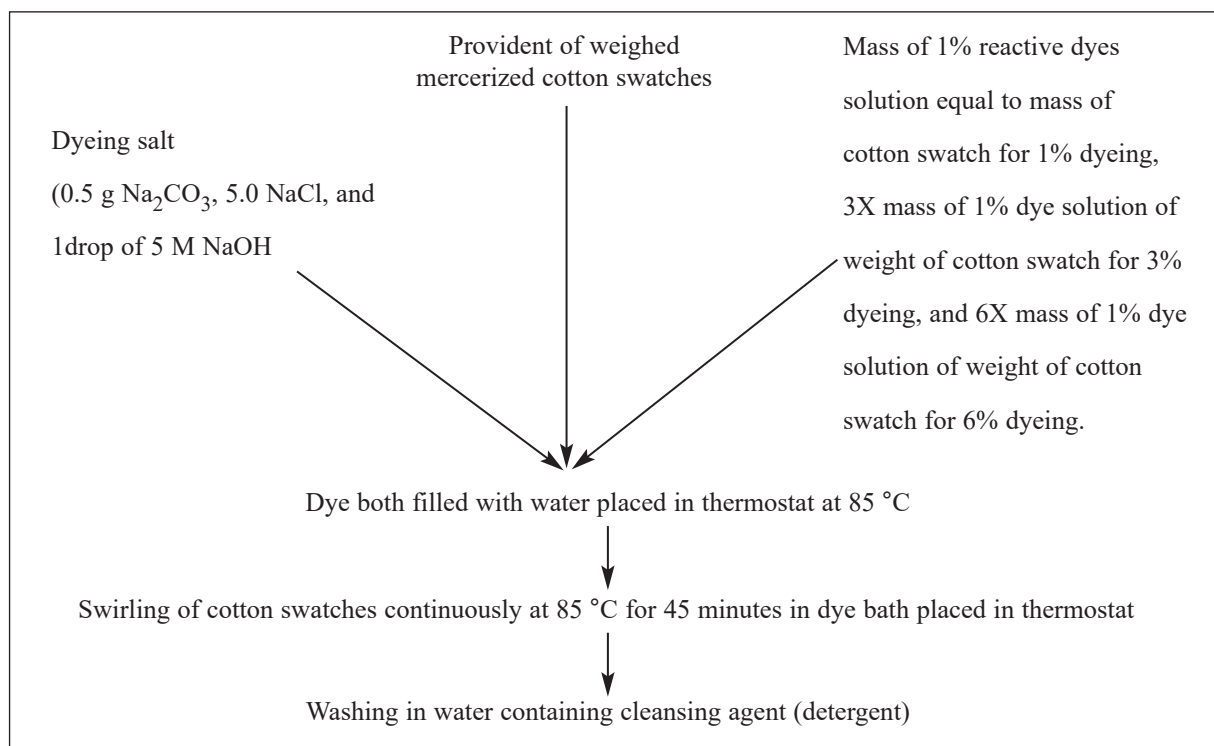


Fig. 1. Dyeing graph (flow diagram of dyeing procedure)

RESULTS AND DISCUSSION

Five trisazo reactive dyes metal complex reactive dyes have been prepared by applying the universal technique of diazotization, coupling, and metallization, in this current research effort. The coupler employed was resorcinol. Three pairing places have been offered by it in electrophilic substitution reaction. Moreover, diversity of shades and excellent fastness qualities has been shown by metal complex reactive dyes of resorcinol. Resorcinol has been coupled with a diazonium salt, ensuring the development of a new monoazo dye ligand, in this investigation. This dye ligand has been metallized in a direction to advance the fastness properties and to acquire a diversity of shades. Predictably, it will be obliged to native growth of artificial reactive dyestuffs, and exclude overseas importations.

Shade cards interpretation

To signify each colourant, a minute portion of four inches four-sided piece or 15 cm by 15 cm coloured swatch was prepared by cutting dyed cotton cloth as a shade card. Printed the tag of the dyestuff on these shade cards. These were utilized to measure the hue of synthesized trisazo reactive dye product in command to yield textiles that were within the typical Pantone colour equivalent choice, shade cards represented the colour criteria [3]. Colour fastness was restrained distinctly for variations in the hue of the trial sample (colour declining) and stain of an undyed firm that approached into contact with the sample throughout the examination (dye flow) on the grey scales.

Synthesis of 6-Napsa-Resorcinol reactive dye series

In this series of dyes, five samples were prepared by diazotization of 6-napsa, coupling with resorcinol, metallization and further attachment with diazotized vinyl sulphone para ester as a reactive anchor. The first dye was unmetabolized, whereas the others were iron (II), nickel (II), copper (II), and zinc (II) complexes. Figure 2 demonstrates the detail of the synthetic scheme as under.

Characterization of 6-napsa-resorcinol-reactive dye series by UV-Visible Spectrum

Reddish-brown dye with λ_{\max} 440 nm and absorbance 1.68 was un-metallized dye 101 (un-metallized 6-napsa-resorcinol reactive dye). Yellowish-brown dye with λ_{\max} 430 nm and absorbance 1.55 was its iron (II) chelate (dye 102), displaying a hypsochromic shift of 10 nm with a 0.13 hypsochromic effect. Reddish-brown dye (dye 103) with λ_{\max} 450 nm and absorbance 1.80 was nickel (II) chelate of dye ligand 101, presenting a bathochromic shift of 10 nm with 0.12 hyperchromic effect. Tan dye with λ_{\max} 460 nm and absorbance 1.70 was copper (II) chelate (dye 104) of dye 101, with the presentation of the bathochromic shift of 20 nm and 0.02 hyperchromic effect. Reddish-brown dye with λ_{\max} 445 nm and absorbance 1.58 was zinc (II) chelate (dye 105) of dye ligand 101, performing a bathochromic shift of 5 nm and 0.10 hypsochromic effect (figure 3).

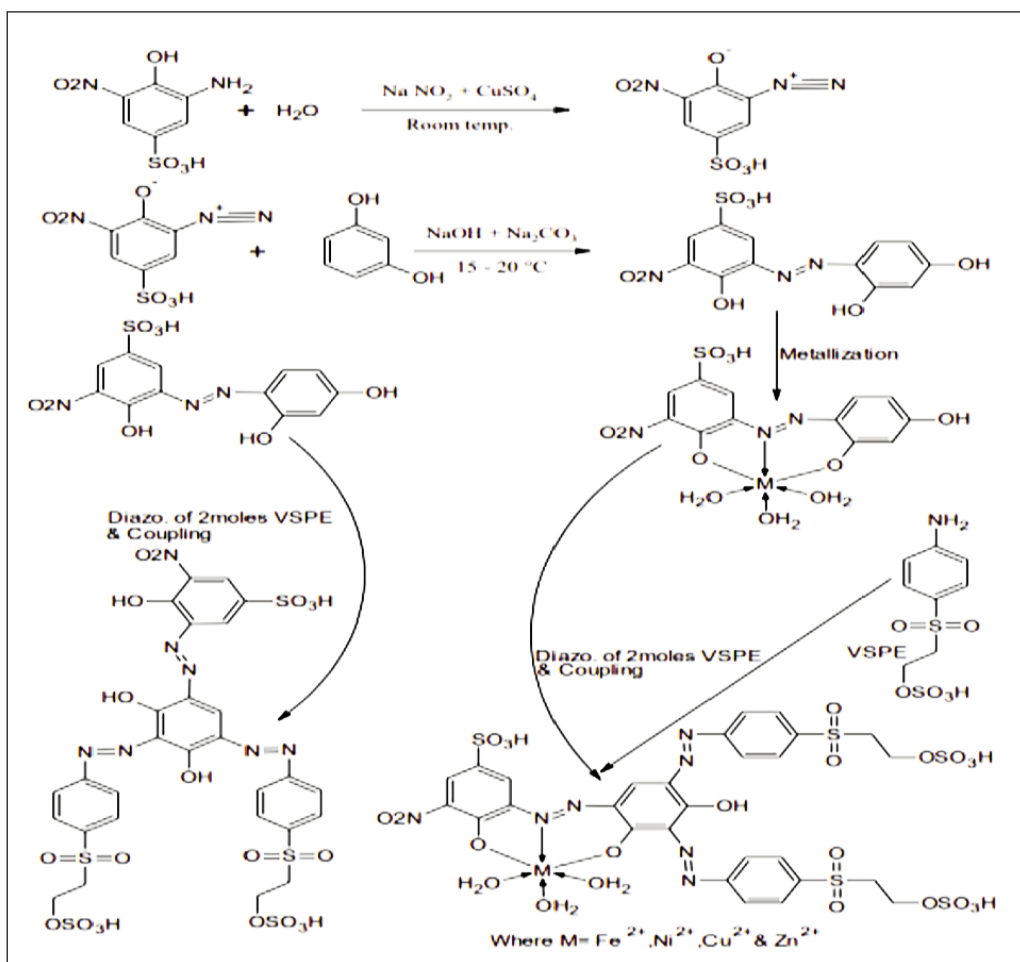


Fig. 2. Scheme of 6-napsa-resorcinol reactive dye series

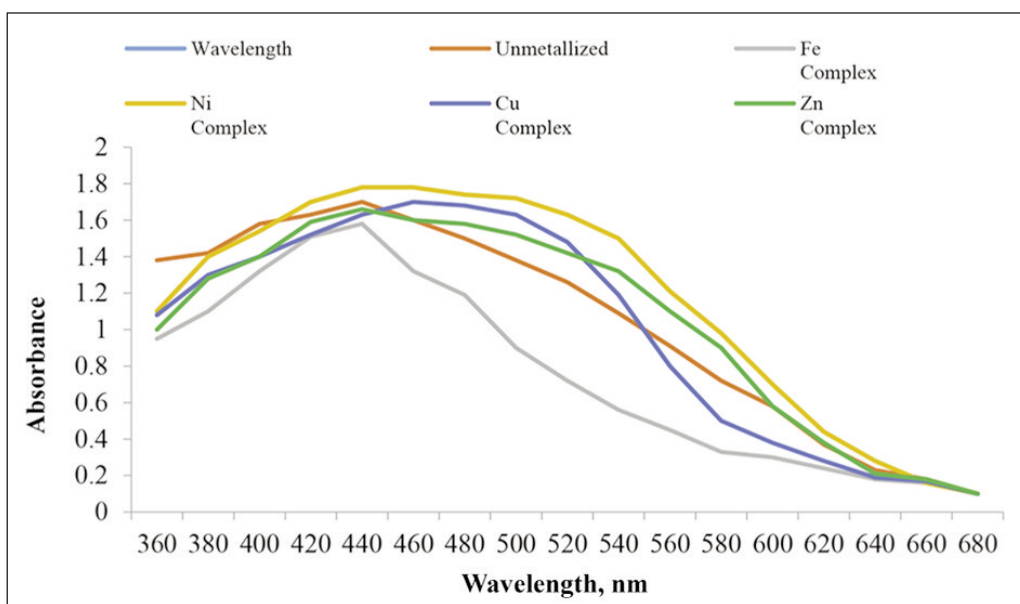


Fig. 3. UV-Visible Spectrum of 6-napsa-resorcinol-reactive dye series

Dyeing procedure

The same dyeing procedure was adopted for the dyeing of 6-napsa reactive dyes as given in the experimental section (figure 1).

Shade cards of 6-napsa-resorcinol reactive dye series

The data given in table 1 is also supported by figure 4, shade card. It is detailed and discussed in the experimental section.

Table 1

FASTNESS PROPERTIES OF SYNTHESIZED REACTIVE DYES		
Dye #	Washing fastness	Light fastness
101	2-3	2-3
102	3-4	4
103	4-5	4-5
104	3-4	4-5
105	2-3	3

The dyed fabric swatches were compared with an internationally recognized Pantone matching system for all shade cards. The un-metallized dye 101 (un-metallized 6-napsa-resorcinol reactive dye) had Pantone matching number 4725 for one per cent shade, 4705 for three per cent shade and 462 for six per cent shade. Dye 102 (6-napsa-resorcinol iron complex reactive dye) had Pantone matching number 4685 for one per cent shade, 4655 for three per cent shade and 464 for six per cent shade. Dye 103 (6-napsa-resorcinol nickel complex reactive dye) had Pantone matching number 4725 for one per cent shade, 1405 for three per cent shade and 161 for six per cent shade. Dye 104 (6-napsa-resorcinol copper complex reactive dye) had Pantone matching number 470 for one per cent shade, 1405 for three per cent shade and 4695 for six per cent shade. Dye 105 (6-napsa-resorcinol zinc complex reactive dye) had Pantone matching number 4745 for one per cent shade, 1405 for three per cent shade and 161 for six per cent shade [1].

FTIR studies of 6-napsa-resorcinol reactive dye series

All detailed features by 6-napsa-resorcinol trisazo fibre reactive dyes were revealed from FTIR Spectral studies. The FTIR spectra of paternal coupler molecule (resorcinol), 6-napsa, un-metallized-6-napsa-resorcinol reactive dye, 6-napsa-resorcinol Fe-complex reactive dye, 6-napsa-resorcinol Ni-complex reactive dye, 6-napsa-resorcinol Cu-complex reactive dye and 6-napsa-resorcinol Zn-complex reactive dye are shown by figure 5, a–g, respectively. The foremost frequencies in FTIR spectrum of 6-napsa are, 3421 cm^{-1} by enolic –OH absorbance, 3090 cm^{-1} by C-H stretching aromatic, 2851 cm^{-1} by N-H stretching, 1649 cm^{-1} C=C stretching, 1591 cm^{-1} C-C (aromatic) stretching, 1031 cm^{-1} by N-O, 1335 cm^{-1} by S=O, 1137 cm^{-1} by SO_3H , 1150 cm^{-1} by C-O stretching, 1145 cm^{-1} by C-N [1]. FTIR spectrum of resorcinol revealed foremost frequencies, 3211 cm^{-1} by enolic –OH, 1590 cm^{-1} by C=C of the aromatic ring, 1495 cm^{-1} by C-C (aromatic) stretching, 1150 cm^{-1} by C-O stretching, 3050 cm^{-1} by C-H stretching [1]. Most of the dyes of this series had all the specific absorptions including N=N in the range of 1410-1580 cm^{-1} and as a demonstrative of this dye series [1]. Un-metallized-6-napsa-resorcinol reactive

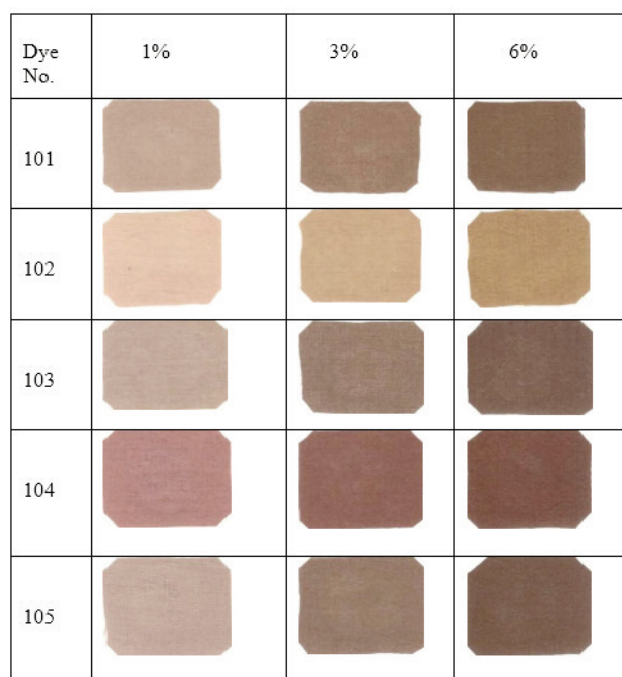


Fig. 4. Shade cards of 6-napsa-resorcinol reactive dye series

dye displayed FTIR absorptions, 3447 cm^{-1} and 3451 cm^{-1} by enolic OH, and NH stretching, 3010 cm^{-1} by C-H stretching aromatic, 1675 cm^{-1} by C-C (aromatic) stretching and 1520 cm^{-1} by NH bending, 1420 cm^{-1} by N=N stretching and bending, 1070 cm^{-1} by N-O, 1137 cm^{-1} by C-O stretching, 1189 cm^{-1} by C-N stretching, and 1325 cm^{-1} by S=O [1]. 6-napsa-resorcinol Fe-complex reactive dye showed FTIR absorptions, 3423 cm^{-1} by water, enolic OH and NH stretching, 2933 cm^{-1} by (C-H aromatic) stretching, 1593 cm^{-1} by C=C (aromatic) stretching and NH bending, 1474 cm^{-1} by N=N stretching and bending by 1446 cm^{-1} , 1218 cm^{-1} by SO_3H , 1135 cm^{-1} by C-O stretching, 1046 cm^{-1} by N-O, 1148 cm^{-1} C-N stretching, and 1350 cm^{-1} by S=O [3]. 6-napsa-resorcinol Ni-complex reactive dye exhibited wide-ranging FTIR absorptions, 3570 cm^{-1} by water, enolic OH and NH stretching, 3080 cm^{-1} by (C-H aromatic) stretching, 1597 cm^{-1} by C-C (aromatic) stretching and NH bending, 1515 cm^{-1} by N=N stretching, and 1446 cm^{-1} bending, 1028 cm^{-1} by N-O, 1350 cm^{-1} by S=O, 1138 cm^{-1} by C-O stretching, 1145 cm^{-1} by C-N stretching, and 1179 cm^{-1} by SO_3H [24]. 6-napsa-resorcinol Cu-complex reactive dye presented extensive FTIR absorptions, 3421 cm^{-1} owing to water, enolic OH and NH stretching, 3080 cm^{-1} by (C-H aromatic) stretching, 1589 cm^{-1} by C=C (aromatic) stretching and NH bending, 1459 cm^{-1} by N=N stretching, and 1459 cm^{-1} bending, 1028 cm^{-1} by N-O, 1350 cm^{-1} by S=O, 1133 cm^{-1} by SO_3H , 1137 cm^{-1} by C-O stretching, and 1143 cm^{-1} by C-N stretching [1]. 6-napsa-resorcinol Zn-complex reactive dye offered widespread FTIR absorptions, 3457 cm^{-1} owed to water, enolic OH and NH stretching, 2930 cm^{-1} by (C-H aromatic) stretching, 1593 cm^{-1} by C=C (aromatic) stretching and NH bending, 1507

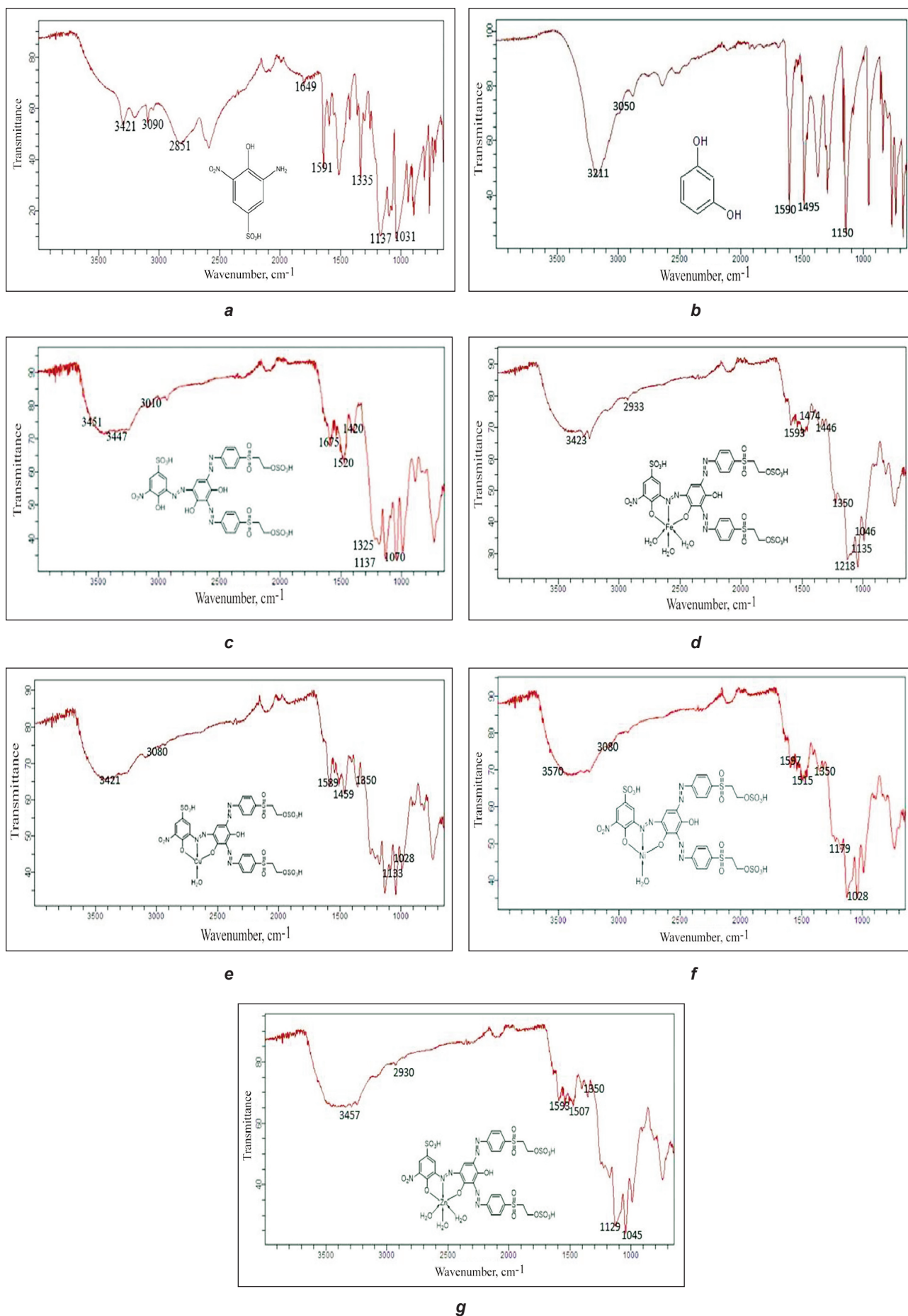


Fig. 5. FTIR spectra: *a* – 6-napsa; *b* – Resorcinol; *c* – un-metallized-6-napsa-resorcinol reactive dye; *d* – 6-napsa-resorcinol Fe-complex reactive dye; *e* – 6-napsa-resorcinol Ni-complex reactive dye; *f* – 6-napsa-resorcinol Cu-complex reactive dye; *g* – 6-napsa-resorcinol Zn-complex reactive dye

cm^{-1} by N=N stretching and bending, 1350 cm^{-1} by S=O, 1129 cm^{-1} by SO_3H , NO_2 , 1045 cm^{-1} by C-O stretching, and 1145 cm^{-1} by C-N stretching [1].

Dyeing results of synthesized reactive dye series

The reactive dyes created in this current study were coloured to mercerized cotton cloths. The dyeing procedure has already been discussed in the experimental section. The reactive dyes created from the new derivatization of resorcinol were confirmed for their characteristics by testing coloured cotton cloths. These properties comprised, washing fastness and light fastness. The values are given in table 1. Shade cards, figure 4, show dyeing results [10].

Dyeing properties of synthesized reactive dyes

Reactive dyes created from the new derivatization of resorcinol with advanced colouring properties have been discovered to be very gorgeous. The wash fastness and light fastness were found to have high values of 4–5 on the grey scales, and 4–5 on the blue scales respectively by maximum metal chelate trisazo reactive dyes. 6-napsa-resorcinol copper chelate trisazo reactive dye manifested the highest values of wash fastness, and light fastness, among all 6-napsa-resorcinol metal chelates trisazo reactive dyes, and established itself at the top one for its dyeing properties. The un-metalized 6-napsa-resorcinol trisazo reactive dye ligand revealed minimal values of wash fastness, and light fastness, as expected due to the presence of free hydroxyl groups. Metal chelates formation of 6-napsa-resorcinol trisazo reactive dyes ligand enhanced the entire dyeing properties like wash fastness, and light fastness,

substantially. The colouring characteristics of reactive dyes made from the new derivatization of resorcinol are specified in table 1.

CONCLUSIONS

From the unique derivatization of resorcinol, a novel azo reactive dye ligand and its Fe^{2+} , Ni^{2+} , Cu^{2+} , and Zn^{2+} complexes were created, and their structures were characterized. The spectroscopic studies have revealed the structure of the unmetallized dye ligand and its metal complexes. These reactive dyes have been defended by achieving high exhaustion and fixation values. The structures of the chelates generated between the trisazo reactive dye ligand and metal ions (Fe^{2+} , Ni^{2+} , Cu^{2+} , and Zn^{2+}) can be clarified using the information obtained from this investigation, as shown in figure 2. Finally, in the dyeing of cotton fabric, all newly synthesized reactive dyes evaluated performed flawlessly. In the future, we will work to improve the dyeing specifications to enhance the quality of these dyes in corporate dyeing procedures. Reactive dyes made from the new derivatization of resorcinol are intended to replace imports from other countries and aid in the development of cost-effective and economical synthetic reactive dyes in Pakistan.

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Authors:

MUREED ABBAS¹, SHUMAILA KIRAN¹, GHULAM HUSSAIN², TAHSIN GULZAR¹, MIRZA NADEEM AHMAD¹

¹Department of Applied Chemistry, Government College University, Faisalabad, Pakistan
e-mail: mureedabbas565@gmail.com; tahsingulzar1@yahoo.com; pioneeravian@hotmail.com

²Department of Chemistry Government Postgraduate College, Jhang, Pakistan
e-mail: ghussain98@gmail.com

Corresponding author:

SHUMAILA KIRAN
e-mail: shumaila.asimch@gmail.com