Synthesis and Characterization of 4,4-Diamino Biphenyl Stilbene-2,2-Disulphonic Acid Based Reactive Dye

Noor Muhammad

Abstract—Reactive dyes were initially introduced commercially for application to cellulosic fibers and this was still their most important use. The growth rate of reactive dyes for cellulosic fibers was expected to continue increasing. The present research was related to synthesize of new 4,4-diamino biphenyl stilbene -2,2-disulphonic acid based reactive dye. The reactive dye was synthesized via condensation and coupling processes. The structural characterization of synthesized 4,4-diamino biphenyl stilbene -2,2-disulphonic acid based reactive dye was done with fourier transform infrared spectroscopy (FT-IR). The synthesized dye had been applied on cotton fabric by exhaust process. Dyed fabric characteristics also had been studied such as light fastness, rubbing fastness, washing fastness and stability to heat treatment.

Index Terms— condensation and coupling processes, fourier transform infrared spectroscopy, exhaust process, light fastness, rubbing fastness, washing fastness and stability to heat treatment.

1 INTRODUCTION

BEFORE all else, colors were gotten from actually happening sources (creatures, vegetables and minerals) e.g. tyrian purple, cochineal, madder, indigo, Prussian blue and ultramarine. The specialty of coloring was known in China since 3000 BC. Fiber responsive colors comprise of a particle having labile gathering. It can frame stable covalent bonds with cellulose [1]. The quick utilization of fiber receptive colors has two fundamental explanations behind cellulosic material. In the first place, it has stable application on texture with ease. Besides, these colors have agreeable quickness to wet treatment because of covalent holding amongst texture and color particles [2].

Colors are hued mixes which are used for passing on shading to the, silk, sustenance stuffs, materials, fleece and so on. A color is a characteristic intensify that can take up some wavelength of the bar dropping on it. The staying of the bar is reflected. The reflected light will at last contain shading adjusting to that of the retained. A color may dismantle up every single detectable pillar from one wavelength which might be reflected. The color will contain shade of the reflected wavelength [3].

Current advances in azo receptive colors science by and large decreases by using of heterocyclic ring plan as diazonium parts that made an intelligent bathochromic move, splendor and improved speed attributes as adjusted to their carbocyclic similar [4]. On this establishment new design of responsive colors has been acquired from quinazoline ring plan. It has been demonstrated that colors upheld on 4-ketoquinazoline moiety have great speed qualities and high steadiness [5]. Colors bolstered on quinazoline ring are of particular noteworthiness as colors have hoisted illustrative impact and great speed attributes of certain cotton materials [6].

- *Co-author, Fatima Saleem,Govertment College University, Faisalabad, Pakistan.PH # +923366303163.*
- *Co-author, Ghulam Abbas,Government College University, Faisalabad,Pakistan, PH #+923346221760.*

2 MAMTERIAL AND METHOD

The union, concoction portrayal and corruption of orchestrated 4,4-diamino biphenyl stilbene - 2,2-disulphonic acid reactive dye was talked about here. The combination of colors was finished in Harris dyes and pigments in Faisalabad. The diverse synthetic portrayal and examination were finished in a joint effort with Pakistan Council Scientific and Industrial Research (PCSIR) labs complex Lahore.

2.1 Equipments

Every single business reagent and solvents required in the blend were of scientific review and were used as gotten. Strong crude materials utilized for the union of colors were taken from Harris dyes and pigments private limited Faisalabad. UV noticeable spectrophotometer (CE-7200) was utilized to discover λ_{max} for recently arranged colors.

2.2 Experimental Devices

Advanced pH meter (Hanna 9818).

Perkin Elmer Lambda 25 UV noticeable twofold shaft spectrophotometer (CE-7200) was used at radiation science lab, division of science and natural chemistry, college of agribusiness Faisalabad.

U-2001 Schimadzu (Japan) fourier change infrared spectrometer (FT-IR) was utilized at Pakistan chamber for scienticfic and mechanical research (PCSIR) Lahore.

Lab scale deplete coloring machine was utilized for the coloring procedure.

2.3 Chemicals

Na_2CO_3, HCl, sodium nitrite, cynuric chloride, 4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive, 1-amino-8-hydroxynapthalene-3,6-disulphonic corrosive, vinylsulfone paraester and 2,4,6-trichloro-1,3,5-triazine

2.4 Method to Synthesize Dyes

A dye was set up by 4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive. The dye N-1 was set up in 1000 ml measuring utencil containing 50 ml water. The color N-1 had been incorporated by taking after the diverse course with distinction of coupling and buildup parts. Biting the dust and fumes strategy are utilized [7]. Mechanical portrayal is concentrated like quick-

ness per modern models society of dyers and colourist.

2.5 Synthesis Route for Dye N-1

2.5.1 Condensation

In the suspension of 0.04 mole of 2,4,6-trichloro-1,3,5-triazine (cynuric corrosive 4g/25mL), 50 mL arrangement 0.02 mole of 4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive (80%) was poured drop astute. The response blend was consolidated at consistent mixing for 1.5 hours at response states of pH 1.5-2 and temperature 0 °C-5 °C. To start with buildup was inspected chromatographically on Whattman channel paper No. 1 by utilizing 2% sodium chloride arrangement as eluent. At that point, N,N-dimethyl benzaldehyde was splashed. The consolidated item was sifted. 100 mL of 1-amino-8-hydroxynapthalene-3,6-disulphonic corrosive (0.04 moles and pH 7) was added to consolidated item without a moment's delay. This blend was warmed gradually up to 45oC and was mixed for 60 minutes for the fulfillment of second buildup at pH 6-6.5. The pH was kept up by 20% Na_2CO_3 arrangement.

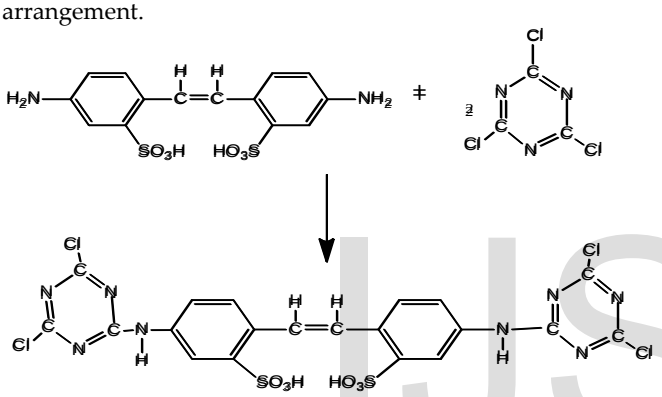

Figure 2.1 Condensation of 2,4,6-trichloro-1,3,5-triazine (cynuric acid) with 4,4-diamino biphenyl stilbene -2,2-disulphonic acid

2.5.2 Condensation of Product Obtained in Article 3.6.1 with 1-amino-8-hydroxy naphthalene-3,6-disulphonic Acid (H-acid)

100 mL of 1-amino-8-hydroxynapthalene-3,6-disulphonic corrosive (0.04 moles and pH 7) was added to consolidated item acquired in article 3.6.2 without a moment's delay. This blend was warmed gradually up to 45 °C and was mixed for 60 minutes for the fulfillment of second buildup at pH 6-6.5. The pH was kept up by 20% Na_2CO_3 arrangement. The response blend was again warmed up to 95 °C-100 °C with modifying pH at 11-12 and mixed for 60 minutes. The subsequent consolidated item was again analyzed chromatographically changing pH 7 by including little measure of hydrochloric corrosive (33% immaculate HCl).

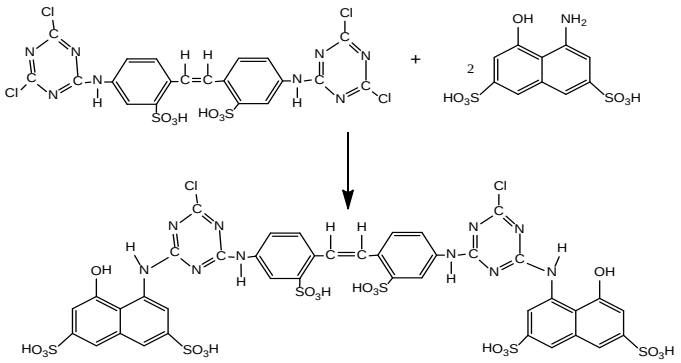

Figure 2.2 Condensation of Product Obtained in Article 2.6.1 with 1-amino-8-hydroxy naphthalene-3,6-disulphonic Acid (H-acid)

2.5.3 Diazotization

Diazotization was completed by dissolving 200 mL H_2O and vinyl paraester (95% immaculate and 0.04 moles) into 150 mL of hydrochloric corrosive (33% unadulterated). The response blend was additionally mixed for 60 minutes at 0 °C. 50 mL of 0.04 mole of sodium nitrite arrangement was emptied drop insightful into vinyl paraester response blend and mixed for 1.5 hours. The diazotized item arrangement was checked with iodine starch pointer paper little amount of sulfamic corrosive ($H^{3+}NSO^{3-}$) was likewise added to the diazotized item.

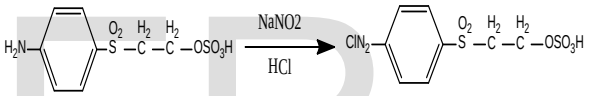

Figure 2.3 Diazotization of vinyl paraester with hydrochloric acid and sodium nitrite

2.5.4 Coupling

The final condensed product was coupled with diazotized product by intensive stirring at reaction conditions pH 7 and temperature 10°C-15°C. After that the synthesized dye was dried in oven at 80°C-90°C.

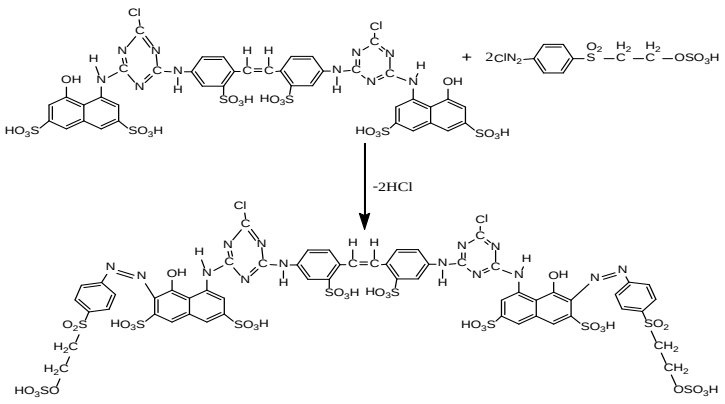

Figure 2.4 Coupling of diazotized vinyl paraester formed in article 2.6.3 with product obtained in article 2.6.2

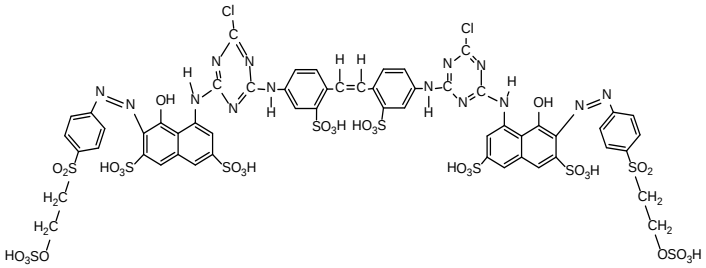

Figure 2.5 Chemical structure of synthesized dye N-1 obtained through condensation, diazotization and coupling processes.

3 RESULTS AND DISCUSSION

3.1 Fourier Transform Infra Red Spectroscopic (FT-IR) Studies for Synthesized Reactive Dyes

FT-IR spectroscopy is most imperative scientific strategy utilized for the assurance of structure and recognizable proof of utilitarian gathering of mixes. Profiles of Fourier change infra red spectroscopy (FT-IR) in strong states for color N-1 lie in the locale of 4000-400 cm-1 which exhibited particular pinnacles appeared in the figure 4.1. Appearance of trademark frequencies of 4,4-diamino biphenyl stilbene-2,2-disulphonic corrosive based responsive colors as for various utilitarian gathering present in them are uncovered specifically locale. Trademark groups for color N-1 at extending recurrence are talked about here. The N=N recurrence in plane showed up in the scope of 1496.97 cm-1. Though, the pinnacles recorded at 1040 cm-1 and 1106 cm-1 were doled out for –OSO_3H extending. The –SO_2 extending band is in the district of 1130 cm-1. Different substituted benzene rings demonstrate absorbance band in the district of 991 cm-1 and 815 cm-1 as appeared in figure 3.1. The extending and bowing frequencies of trademark utilitarian gatherings are condensed in the table 3.1.

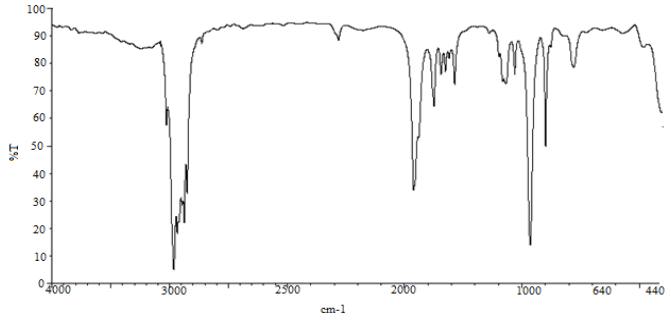

Figure 3.1 FT-IR spectrum of synthesized reactive dye N-1

Table: 3.1 Stretching and bending frequencies of characteristic functional groups present in dye N-1.

Characteristic peak (cm^{-1})	Assignment	Functional group
1496.97	N=N stretching	-N=N-
1040, 1106	-OSO_3H stretching	-OSO_3H
1130	-S=O stretching	-SO_2
991, 815.7	Substituted benzene rings	Benzene rings
3079	C=C stretching	-C=C-

3.2 Application of Prepared Dye by Exhaust Method

In weariness technique, a chose amount of material is included into the fumes coloring machine and with specific coloring time, it conveys to harmony with color arrangement. This strategy includes assimilation and desorption of colors from color shower because of substantively of the chemicals to the material substrate. For the most part, fumes technique is tedious and require a considerable measure of water and vitality.

3.3 Result of Salt Concentration on Exhaust Dyeing

Salt is vital to build the rate and reach out of fumes coloring. By utilizing diverse convergences of salt, the consequences of shading quality were analyzed for two 4,4-diamino biphenyl stilbene -2,2-disulphonic corrosive based reactive dye. Salt particle, for example, sodium chloride breaks to Na^+ and Cl^- particles in water. A Na^+ particle kills outskirt between color atom and fiber particles. It additionally encourages the rate of weariness of color to cellulosic fiber. Na^+ particles are related with water atoms and breaking point the degree of hydrolysis of color particles. The impact of salt is noted in various fixations i.e. 2, 4, 6 and 8 g regarding (%) shading quality qualities to upgrade response conditions for one recently arranged cellulosic receptive color. The qualities acquired for cellulosic materials clarified most extreme shading quality (%) in the scope of 70-80 %. The shading quality abatements with increment in salt focus over the ideal esteem. On the off chance that we utilize surplus electrolyte, it might bring about the gathering of color which brings about bringing down the movement rate. It prompts unlevel of coloring on cellulosic material [8]. 8 g of salt focus was resolved in the weariness investigation of color N-1. The shading quality was improved for the prepared color. On the off chance that salt fixation is higher than ideal focus; it may bring about color affidavit on cellulosic material, which prompt uneven coloring. In the event that salt focus is not as much as ideal fixation, it may bring about poor obsession with cellulosic material. Its outcomes for color N-1 are appeared in the figure 3.2.

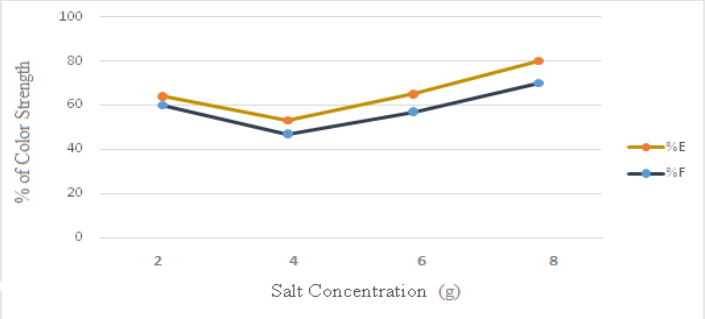

Figure 3.2 Effect of exhaustion (%E) and fixation (%F) values at different salt concentration for dye N-1.

3.4 Result of Dyeing Time on Color Strength of Cellulosic Material

In coloring process, cellulosic materials are hued forever with a color in a homogeneous way to acquire great shade. This procedure is conveyed in fluid arrangement. Time is extremely powerful parameter for checking exhaustion (%E) and fixation (%F) prepare on cellulosic fiber. The fumes coloring procedure was considered for various time interim i.e. 40, 50, 60 and 70 minutes. Its point was to check most extreme shading quality on cellulosic material. The rate of dispersion of color happens through the col-

or shower on cellulosic surface and inside of fiber. The pattern of exhaustion (%E) and fixation (%F) was improved with coloring time. The greatest shading quality of weariness for orchestrated dye N-1 84 %. Comes about got clarified that 70 minutes of coloring procedure was sufficient for greatest fatigue rate. It is actuality that additional time is vital for color to move into the inside of cellulosic material. The color atoms additionally require enough time to join with hydroxyl gathering of cellulosic material. For brief time i.e. 40 minutes, colors speak to less % weariness values as adjusted with colored cellulosic material acquired at higher coloring time of 70 minutes. Its outcomes for dye N-1 appeared in the figure 3.3.

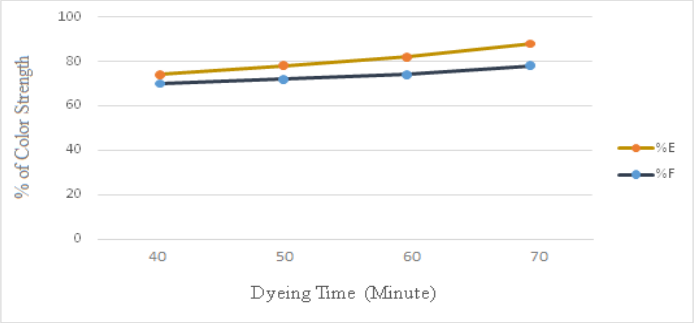

Figure 3.3 Effect of exhaustion (%E) and fixation (%F) values at different dyeing times for dye N-1.

3.5 Result of Temperature on Dyeing Process

The rate of coloring procedure is influenced by temperature of color shower arrangement. To get ideal weariness and color take-up, it is exceptionally important to check and keep up the color shower temperature. The temperature of coloring procedure changes from color to color for ideal fascination and it ought to be kept up. In the coloring procedure, the atoms of color move into the cellulosic fiber. The rate of dissemination of color atoms all through substrate increments with increment in temperature of color shower. It has been watched that when temperature expands, porosity of cellulosic material likewise increments. Along these lines, surface range of cellulosic material upgrades and makes more space for color particle to ingest into the substrate i.e. cellulosic material. The accompanying figure 4.4 demonstrates the after effect of coloring temperature for two arranged receptive colors on cotton material. These analyses were performed on temperatures 50 oC, 60 oC, 70 oC and 80 oC. The fumes coloring procedure was utilized on cotton material with 2 % shade for both the colors. The % of fumes coloring upgrades with increment in coloring temperature for both blended receptive colors. At higher temperature, cotton fiber opens and enables the color particles to exchange effortlessly. The color particles are joined to hydroxyl gathering of cellulosic cotton material. In this manner, color particles enter the fiber [9]. The % of depletion increments with increment in temperature and it is as per motor hypothesis. Dynamic hypothesis expresses that rate of concoction response improves with increment in temperature. The colors utilized as a part of this exploration work demonstrated top of the line obsession and weariness values at 70 oC and 80 oC. The prepared dye appeared more than 60-70 % exhaustion values. The % fixation (%F) of was likewise noted. It was additionally 60-70 %.

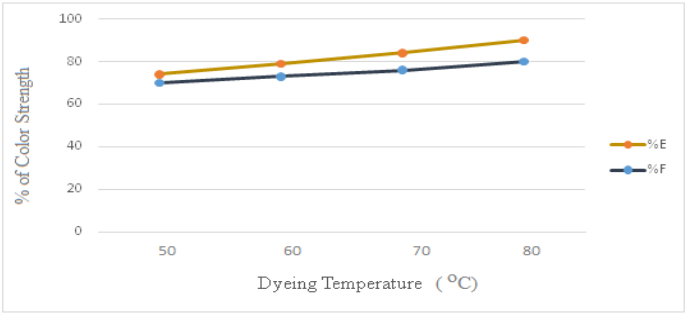

Figure 3.4 Effect of exhaustion (%E) and fixation (%F) values at different dyeing temperatures for dye N-1.

3.6 Consequence of pH

The impact of pH was checked for two arranged receptive colors on cotton material. Its outcomes are appeared in the figure 4.5. Cotton material were colored at four different pH values i.e. 8 9, 10 and 11 individually. It was seen that color take-up diminished obviously as pH of color shower expanded. It was additionally watched that shade of shade of colored cellulosic material ended up noticeably lighter with upgrading pH. The high pH was awful for depletion rate and in addition obsession rate of colors on cellulosic fiber. Its reason is that as pH improves, finish ionization happens for hydroxyl gatherings. Hydroxyl bunches begin to lose their protons. Therefore, adsorption of prepared receptive colors was seen in lesser rate at high pH. This kind of results was gotten from the investigation of exhaustion (%E) and fixation (%F) rates at different pH values. The most positive depletion and obsession rates were acquired when color was connected at pH 11. For the advancement of pH for both 4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive based receptive color was noted at different pH regarding exhaustion (%E) and fixation (%F) comes about [10]. Greatest color shower depletion esteem was 78 % for color N-1 at pH 10. At the point when pH is additionally expanded, % depletion rate diminishes. The take-up of both the incorporated receptive colors in coloring cellulosic material was in the scope of 60-80 %. At the point when pH is low, the quantity of anionic charges on cellulosic material increments. Along these lines, color atoms indicate greater partiality on the cotton texture. The holding component between receptive color atom and cellulosic fiber is covalent linkage. At the point when pH is high, hydroxyl gathering will be totally ionized. At the point when the pH is low, there is an improving improvement in color take-up which is normally ensured to color adsorption on cotton textures.

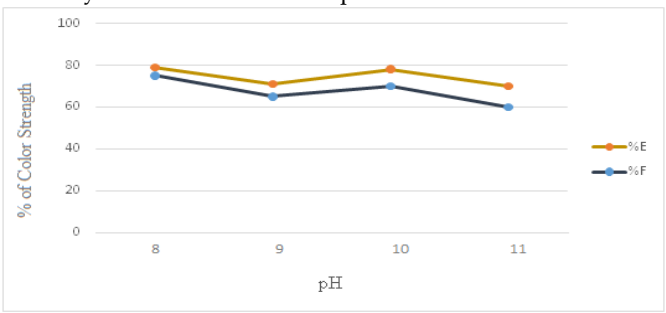

Figure 3.5 Effect of exhaustion (%E) and fixation (%F) values at different pH for dye N-1.

3.7 Fastness Properties of Dyed Fabrics

Shading fastnesses to light, sweat, washing, chlorinated water, cleaning and crocking were assessed of ISO (International Organization for Standardization) standard speed test convention. With coloring quality parameters, for example, salt focus, pH, Temperature and coloring time; cotton texture gave sufficient shading quickness properties. The recoloring occurring on the colored cotton was evaluated onto the standard dark scale: 1 remains for poor, 2 is for reasonable, 3 for direct, 4 is for good and 5 is for incredible. Comes about got were assessed by methods for ISO test.

The speed properties like crocking, light, chlorinated water, sweat and quickness to washing were resolved utilizing ISO standard speed test convention. The adjustment in shading shade discovered outwardly by utilizing dim scale. The best quickness acquired by fumes strategy. These outcomes are appeared in the accompanying table 4.1. The qualities for shading quickness to washing and crocking (dry and wet) were in the arrangement of 4-5. This range for shading quickness was of good quality. Wash speed evaluations of both responsive colors were likewise noted by utilizing ISO test 105CO6/C2S. This test was additionally palatable at each profundity of shade. Speed test grades of incorporated responsive colors were decided for chlorinated water, light and sweat acceptable (4-5) [11]. The better speed properties were showed up because of responsive nature of arranged colors. These colors have more noteworthy capacity to settle on the cellulosic fiber in the wake of washing and drying process. Reactive dyes have great quickness properties because of covalent nature [12].

Table 3.1 Fastness properties of two synthesized dyes on cotton fabric by exhaust dyeing process.

Fastness properties	Dye N-1
Crocking fastness	4-5
Perspiration fastness	4
Dry cleaning fastness	4
Washing fastness	4.5
Light fastness	4
Chlorinated fastness	4

4 CONCLUSION

4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive based receptive color (one) were combined by utilizing back to back strides of buildup, diazotization and coupling responses. In these three stages, coupling parts were H-corrosive (1-amino-8-hydroxynaphthalene-3,6-disulphonic corrosive), J-corrosive (6-amino-1-naphthol-3-sulphonic corrosive) and gamma-corrosive (6-amino-1-naphthol-4-sulphonic corrosive) individually. 4,4-diamino biphenyl stilbene - 2,2-disulphonic bunches had been unequivocally appended as the primary responsive framework for cellulosic materials. The shade of arranged colors adjusted with acids i.e. with H-corrosive are purple and with J-corrosive are orange to red. 4,4-diamino biphenyl stilbene - 2,2-disulphonic corrosive based receptive colors were chosen and orchestrated in light of splendid shades, brilliant speed properties and expansive applications. The synthetic structure and a few spectroscopic components of arranged azo responsive color was dictated by utilizing systematic strategies like FT-IR subsequent to applying physical and compound refinement technique to the color. General adequate subjective concurrences with spectroscopic information were accomplished.

One of the first destinations of this exploration work was to check distinctive coloring circumstances to watch their absorbance propensity towards cotton materials. prepared color was connected with 3 % color arrangements on factory scoured, dyed and desized cellulosic materials. Their application attributes were likewise contemplated. The depletion system was used for coloring cotton textures. These methods force color atoms inside the texture for better dissemination and evacuates abundance color. Profound shades were accomplished by utilizing weariness coloring technique at appropriate coloring conditions i.e. 70-80 %. It demonstrates that colors set up well on cellulosic textures. The level of exhaustion (%E) and fixation (%F) of both the colors on cotton material was contemplated and get satisfactory % E/% F values because of their high substantively.

The shading quickness pictured by standard dark scale and other physico-concoction test, both arranged colors were very adequate and better in examination with the related business colors. Distinctive quickness attributes like crocking, chlorinated water, sweat, light, washing and cleaning were assessed utilizing ISO tests conventions, which showed comes about with the assistance of dark scale utilized for assessment.

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