

# Synthesis and Application of Novel Reactive Dyes for Cotton Fabric with Enhanced Properties

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# Abstract

The consumption of inorganic electrolytes in typical existing methods of reactive dyeing of cotton contributes to the chemical pollution in textile effluents and motivates the development of reactive dyes capable of producing effective salt free coloration. This study involves the development of novel azo-based reactive dyes containing a monochlorotriazine reactive group and choline-derived functionality. The dye was synthesized and investigated for salt-free dyeing of 100% cotton fabric. Ortho- and para positional dye structures were synthesized through consecutive diazotization, azo-coupling, and nucleophilic replacement reactions and applied at (0.5–4% o.w.f.) under controlled conditions of 60°C for 60 min at pH 10.8–11.2 using sodium carbonate as the alkali, without the addition of inorganic salt. Following dyeing, the fabrics were treated at 90°C with commercially available detergent and subjected to three consecutive hot washing cycles to remove unfixed dye. FTIR and UV–visible spectroscopy confirmed the characteristic functional and chromophoric features of the synthesized dyes. Dyeing considerably improved the cotton fabric from ultraviolet radiation, enhancing UPF from approximately 7 to 40 with increasing dye concentrations, meanwhile UVA and UVB blockage up to 90%. The dyed fabric demonstrated outstanding dry rubbing  $\approx 4.5$ , good to excellent wet rubbing 4.0–4.5, and satisfactory washing fastness, aiding efficient dye absorption and fixation without addition of salt as an electrolyte in dyeing process. The ortho derivative showed comparatively better light and washing fastness, whereas the para derivative provided slightly higher UV protection. No detectable antibacterial activity against *Staphylococcus aureus* was observed under the applied agar-diffusion conditions. The findings demonstrate the potential of the synthesized choline-functionalized monochlorotriazine dyes as salt-free reactive colorants for producing durable and ecofriendly and sustainable cotton textiles by removing salt consumption, providing a promising approach for reducing electrolyte use in conventional reactive dyeing.

## 1. Introduction

Fiber reactive dyes are termed as reactive dyes. These dyestuffs are colorful organic compounds that can give rise to a covalent linkage formation among reactive sites of the colorant molecule and nucleophilic groups present in molecular chains of polymer within the structure of fiber [1]. As a result of the covalent dye polymer connections, the dyes become a chemical component of the fiber. Covalent linkage between dye and polymer are generated with hydroxyl groups ( $-\text{OH}$ ) of cellulose, amino ( $-\text{NH}_2$ ) hydroxyl, and ( $-\text{SH}$ ) marcapto groups present in proteins, and ( $-\text{NH}_2$ ) amino sites present in polyamides [2]. Surface physical adsorption or mechanical holdings onto the surface of the cloth characterizes all general groups of synthetic dyes, resulting in major difficulties such as poor wash-fastness. Researchers have long wished for the formation of a covalent bond between color and fiber [3].

Reactive dyes are attributed to good wash-fastness because covalent linkages were considered the strongest attractive forces between different molecules. The energy needed for breaking this type of bonding would be comparable to the energy required to break covalent bonds within the fiber [4]. Protein and polyamide fibers have also benefited from the development of reactive dyes. Furthermore, while

researching in the field of production of fiber reactive dyes for polyester fibers and polypropylene fibers has reached the point of technical feasibility, such dyes are not yet commercially viable [5]. Cotton is the most common type among cellulosic fibers. Many other cellulose-based fabrics that can be dyed using cotton dyes include viscose rayon fibers, linen fibers, cupra-ammonium rayon, jute fibers, and lyocell fibers [6].

Fixation of colorant could be approached in two ways, penetrating a colorant molecule within the structure of fiber, and constructing a dye that is covalently reactive towards the fiber. Cross & Bevan in 1895 evaluated the first covalent linkage between dye molecule and fiber [7]. The first approach of producing color within fiber is made successful by converting cellulose into "soda cellulose" by treating strong alkali, which then allowed to react by benzoyl chloride to produce benzoyl cellulose. Nitro group is decreased, and the amino ( $-NH_2$ ) group is diazotized that results in benzoyl cellulose formation. To make "dyed" fibers, the diazonium group was attached with N,Ndimethylaniline [8]. Later, successful attempts made to induce reactive groups in colorant molecules that tend to react with hydroxyl groups of cellulose instead of modification of whole cotton structure. After this successful approach, a large number of dyes were developed not only for cellulosic substrates and for protein fibers like wool and silk and synthetic fibers like nylon and cellulose acetate [9].

A reactive system, chromophoric system, bridging group, and solubilizing systems are the four distinguishing properties of a normal reactive colorant molecule. The reactive system is the basic distinction of reactive dyes from all other dyestuffs [10]. There are many types of reactive systems that are reported in literature but here we will report two reactive systems, namely monofunctional and bifunctional reactive systems. With the nucleophilic groups in the fiber, monofunctional systems can only react once. Examples of such systems refer to halotriazine and vinylsulphone systems. There are two corresponding consumable halogen substituents in the dichlorotriazine, difluoropyrimidine, and dichloroquinoxaline heterocyclic ring systems [11]. The reactivity of the remaining halogen substituent is considerably reduced when anyone of the halogen atoms is shifted by interaction with functional sites in the fiber or with alkali present in the dyeing medium. Bifunctional fiber reactive dyes have two independent reactive centers that react with the fiber's appropriate groups. They can also react chemically with more than one group in the molecule of the fiber chain. Covalent bonds can develop either inside the equivalent polymer chain or between two neighboring chains because of these processes [12]. Techniques like electron microscopy, surface area evaluation, and swelling in cadoxensolvent were utilized to get data that show the production of interconnections between neighboring cellulose chains present in cotton colored with several types of bifunctional reactive colorants. In literature, bifunctional reactive systems are also of two types that are reported [13].

The second main component of dyestuff is its chromophoric system that gives them a distinct colour. To achieve a specified shade area, commercial reactive dyes require the right selection of chromophores. Reactive dyes are increasingly chosen based on shade, according to market data [14]. The groups that connect the reactive scheme to the chromophore are known as bridging groups. While these groups are required for synthetic purposes, they also have a substantial impact on reactivity, degree of fixation,

stability of reactive colorant molecules, and other dyeing characteristics mentioned as substantivity and migration [15]. An amino (-NH-) group is a common bridging group. Bridging groups based on ether and mercapto groups have been investigated, but the bonds are found often unstable. They're also more difficult to create than imino bridging groupings [16]. To a limited extent, groups like carboxamide & sulphonamide are found stable and can be employed in the form of bridge groups. The important features like water solubility, substantivity towards textile substrates, migration rates, and wash off are observed due to solubilizing groups. The sulphonic substituent is the most common solubilizing group in reactive dyes [17].

Synthesis of azoic dyes were become possible after the invention of diazotization mechanism, that mechanism was first defined by Peter Greiss in middle of nineteenth century [18]. Azo reactive dyes demand the diazotization of primary compounds containing amino groups in structure. This reaction is catalyzed by hydrochloric acid and sodium nitrite; it also demands maintenance of very low temperature ranges from 0-5°C. Structural analysis of diazonium salt evaluated that; it shows relatively weak electrophilic properties. So, it demands an electron rich species to attack to materialize electrophilic substitution reaction. The selection of coupler for electrophilic substitution reaction with diazonium salt is a matter of selection by keeping in mind structure property relationship of reactive dyes, most used coupler is H-Acid, J-Acid etc. these are favored due to their higher solubilizing groups present in structure. The last step involved is the attachment of a reactive group to the dye. These reactive sites can be attracted to coupler or primary compounds by simple nucleophilic substitution mechanisms. The mono-azoic chromophore is produced by this whole phenomenon [19]. Next step of synthesis is the attachment of a reactive group on the molecule. This process involves a substitution mechanism between reactive parts of reactive groups such as chlorine atom of cyanuric chloride and hydrogen attached at amino groups of couplers [20].

The sustainable world demands biodegradable textiles. Production of natural fibers provides sites for regulation of atmospheric carbon dioxide [21]. Less energy is required for production of natural fibers as compared to manmade textile fibers. Cotton is an important textile fiber that grows in the upper region of the cotton plant seed. Usually a cotton fiber contains 90–95% cellulose which is a natural organic compound represented by the general formula  $(C_6H_{10}O_5)_n$ . The raw cotton fibers also take waxes, pectins, organic acids and inorganic substances with them, due to which ash is produced when fiber is burnt. Structure of cellulose is linear containing 1, 4-β-D-glucose units [22].

The adsorption and fixation of dyestuff on fabric take place in different steps. Firstly, dye exhaust from the dyeing medium onto the surface of the fiber. Adsorption of dye at the fiber surface is followed by diffusion of dye molecules from the fiber surface into the fiber pores. Process of dye relocation that results in the dye homogeneity [29]. When the dye in the solution and the dye in the fiber reach a state of equilibrium, migration happens. The creation of a covalent link between the dye and the fiber that allows dye to be fixed. Fixation to cellulose occurs primarily in alkaline environments, but fixation to polyamide can occur in moderately acidic environments [30]. Competitive hydrolysis of reactive dye happens during the fixing stage when the dye molecules get attacks on hydroxide ions present in the dyebath. Because

the hydrolyzed dye has diffusion and adsorption properties that are extremely like the original reactive dye, it exhausts onto the fiber surface with the reactive form. Physical forces resembling those of a direct dye on cotton hold hydrolyzed dye adsorbed on the fiber surface in some situations. Lowering the degree of fixation of fiber reactive dyes occurs when they are hydrolyzed [31]. To improve crock-fastness and wash-fastness, unfixed and hydrolyzed dye molecules should have to be separated from the fiber by a washing process after dyeing. Fixation takes precedence over hydrolysis in practice due to several important variables [32].

Chemical reaction takes place in two ways; it can be nucleophilic substitution or nucleophilic addition mechanism. In the first approach of nucleophilic substitution mechanism, the leaving system of reactive system is removed, which produces a link with a nucleophilic site present on the polymer chain. This type of dye fiber fixation is known as nucleophilic substitution. This technique is elaborated by the reaction of a monochlorotriazine reactive dye with a hydroxy group of cellulose and/or an amino group of keratins. During dye application, the competitive hydrolysis interaction between the dyes and water is accounted for using the same procedure [33].

Formally, the dye-fiber reaction can be a nucleophilic addition, which occurs when a nucleophilic group in the fiber adds across an activated carbon-carbon double bond in the reactive group. Vinyl-sulphone moiety is present in most reactive systems. The vinyl-sulphone reactive group is frequently absent from commercial dyes [34]. Reactive dyes containing phosphoric acid as reactive site are fixed to cellulose under lower pH in the presence of cyanamide at a temperature of approximately 200°C. Esterification method is used to carry out the reaction. A competitive hydrolysis reaction does not occur in this scenario [35].

## **2. Materials and method**

### **2.1 Materials**

The following materials and equipment were used: choline chloride, various solvents, pH meter, cyanuric chloride, 100% cotton textile, meta-ester, washing solution, sodium nitrite, sodium carbonate, deionized water, a sequestering agent, ice, sulfamic acid, water baths, beakers, graduated cylinders, magnetic stirrers, and hot plates, and hydrochloric acid (HCl), acetone, and a filtration assembly.

### **2.2 Methodology**

Firstly, dye has been synthesized with procedure mentioned below and after that reactive dye is applied to cellulosic fabric with optimized and fixed concentrations of dye, auxiliaries and liquor ratio.

#### **2.2.1 Dye synthesis procedure**

- **Activation of cyanuric chloride**

Due to its high reactivity, cyanuric chloride (1.85 g, 0.01 mol) was dissolved in acetone at temperatures between 0 and 5°C. The pH was lowered to 3.5 with HCl for optimal activation of the triazine ring for the next nucleophilic substitution. The mixture was stirred for 15 min to get uniform activation. This step makes the triazine ring available for further reaction with other reactants.

- **Coupling with J-Acid**

At pH 7, J-acid (2.39 g, 0.01 mol) was prepared by neutralizing the acidic groups with sodium carbonate, which increases the solubility and reactivity of the J-acid. The J-acid solution was then added dropwise to the cyanuric chloride solution over a period of 30 minutes at 0–5°C to ensure the slow addition of the J-acid solution and minimize side reactions. The reaction mixture was stirred for another 30 minutes at the same temperature for the formation of mono-substituted intermediate. The –NH, provided from J-acid, make covalent bonds with the available triazine ring in this step, forming a stable bond.

- **Reaction with choline chloride**

When the dye formulations consist of one molecule of choline chloride, 1.55 g (0.01 mol) of choline chloride was added to the intermediate solution at room temperature (25°C). Sodium carbonate was used to maintain the pH at 7.3. If two molecules of choline chloride were used in the dye, 3.1 g (0.02 mol) of choline chloride was used. Choline chloride is a reactive modifier which adds a quaternary ammonium group to the dye molecule and thus removes the need for salt in the dyeing process. The reaction mixture was stirred for two hours at room temperature to make sure of the completion of the reaction and heated at 70–75°C for 30 minutes to increase the stability of the dye molecule and the efficiency of the reaction.

The diazonium salt was stabilized by keeping the temperature between 0 and 5°C while dissolving ortho-ester (5.3 g, 0.02 mol) and para-ester (4.02 g, 0.02 mol) separately in a mixture of 50 mL of cold water and 2 mL of hydrochloric acid in both solutions. Sodium nitrite (2 g, 0.02 mol) was added to the solution slowly keeping pH at 4.5 all the time. The mixture was stirred for an hour to obtain the diazonium salt, which is an important intermediate in the preparation of azo dyes. To remove excess nitrous acid, 2 g (0.02 mol) of sulfamic acid was added, and the reaction was said to be complete when there was no further bubbling. The pH was kept in the range of 4.5 to 5.0 and stirring continued for a further hour to ensure the stability of the diazonium salt.

The diazonium salt solution was added dropwise to the dye intermediate solution with the pH kept between 3 and 4 so that the coupling reaction can be accelerated. The pH was then raised slowly to 8.5 in 1 hour to stabilize the formation of an azo bond. The process gave a new salt free reactive dye for dyeing.

- **Diazotization of para and ortho ester**

To stabilize the diazonium salt, 5.3 g of para ester (0.02 M) was dissolved in 50 mL of cold water and 2 mL of hydrochloric acid to keep the temperature between 0 and 5°C. While keeping the pH of the solution

at 4.5, sodium nitrite (2 g, 0.02 mol) was gradually added. The diazonium salt, a crucial step in the synthesis of azo dye, was formed by stirring the mixture for an hour. The addition of sulfamic acid (2 g, 0.02 mol) eliminated the surplus nitrous acid, as seen by the bubbling stopping. To guarantee the stability of the diazonium salt, the pH was kept between 4.5 and 5 and stirred for an additional hour. Following the same process ortho ester solution was also formed by adding 4.02 g (0.02 M)

- **Coupling of diazotized para ester with dye intermediate**

To speed up the coupling reaction, the diazonium salt solution was gradually added to the dye intermediate solution while keeping the pH between 3 and 4. After an hour of stirring, the pH was progressively increased to 8.5 over the course of the following hour to stabilize the formation of azo bonds. The result is a new reactive dye that doesn't contain salt and is prepared for use in dyeing.

- **Salting out the dye**

One mole of sodium chloride was added per liter of solution, to precipitate the dye. Salting out decreases the solubility of the dye in water, which helps to isolate the dye. Precipitated dye was filtered, washed, and dried for future use.

- **Dye application onto cotton fabric**

The synthesized dyes were applied to 100% cotton fabric using a salt-free dyeing process under controlled conditions. Both ortho- and para-position dyes were applied at concentrations of 0.5%, 1%, 2%, and 4% on the weight of fabric (o.w.f.). Dyeing was carried out at 60°C for 60 min at pH 10.8 – 11.2, with sodium carbonate used to adjust and maintain the alkaline conditions. No inorganic salt was added during the dyeing process. Following dyeing, the treated fabrics were subjected to a hot washing treatment at 90°C using commercial detergent as a sequestering/cleaning agent to remove unfixed dye. The dyed samples were subsequently subjected to three consecutive hot washing cycles. The retention of colour after these treatments, together with the subsequent washing and rubbing fastness results, provided supporting evidence of effective dye uptake and fixation on the cotton substrate under salt-free dyeing conditions. Sample dyeing profile is shown in Fig. 2.1.

### 3. Characterization

After the sample preparation, the fabric samples were subjected to a series of standard tests to determine if the cotton fabric had the necessary properties, and to analyze the fabric based on the experimental results.

#### 3.1 Ultraviolet protection factor (UPF)

Test method for measuring the ultraviolet protection factor (UPF) of textiles that measure the percentage of ultraviolet UV-A (400 – 320 nm) and UV-B (320 – 290 nm) radiation transmitted through and blocked by the material following the standard protocol (AATCC 183). A device was used to measure

transmittance over a range of wavelengths. The fabric specimens were cut using the test standard requirements specified in EN 13758-1. The samples were conditioned for a minimum of 24 hours at  $21 \pm 1^\circ\text{C}$  and  $65 \pm 2\%$  relative humidity. Sample placed in a holder that holds the fabric to analyze the UPF.

The percentages of transmittance for UV-A and UV-B were determined. Transmittance values were used to calculate the UV blocking percentage. The mean UPF value and standard deviation (SD) were calculated to determine the protective ability of the fabric. The Ultraviolet Protection Factor (UPF) is a measure of how well a fabric will block out ultraviolet radiation. For instance, a fabric that has a UPF of 50 + allows less than 1/50th (less than 2%) of UV rays to pass through, which means that it blocks more than 98% of the UV radiation. The UPF rating is the degree to which UV rays will pass through the material. A UPF of 50 + is considered "excellent UV protection" and is best for minimizing the effects of UV damage to skin. Fabrics that have a UPF rating of 50 + are especially appropriate for outdoor activities in high sunlight environments.

## 3.2 Antimicrobial activity

The antimicrobial test (AATCC TM147) qualitatively assesses the antibacterial activity of treated textile samples. This test is used to assess the ability of a material to reduce the possibility of microbial contamination and the ability of the material to inhibit bacterial growth. Nutrient agar powder was dissolved in distilled water according to the manufacturer's instructions. The medium was sterilized by autoclaving at  $121^\circ\text{C}$  for 15 minutes. Nutrient agar was then sterilized and poured into petri dishes, which were allowed to cool and solidify. Standard bacterial strain such as *Staphylococcus aureus* (S. aureus) ATCC 6538. The Petri dishes were left for 24 hours at  $37^\circ\text{C}$ . The growth of the bacteria on and around the plates was then checked. Sterilized fabric samples were placed next to the streaked bacterial line on the nutrient agar plate. A loopful of the test microorganism was streaked in one line across the middle of the agar plate.

## 3.3 Rubbing fastness test

The rubbing fastness is a measure of a fabric's ability to resist colour transfer when rubbed against another surface. This test checks the quality of the dyeing process and helps ensure the dye durability in daily use, especially in areas where friction may occur. Standard pressure setting/weights were set as defined by ISO 105 X12. The rubbing finger (round or rectangular pad) was covered with a dry white cotton rubbing cloth. A piece of dyed fabric was firmly attached to the crock meter platform. The rubbing finger was used at the standard pressure, and 10 complete rubs (to and fro) were given. The white cotton cloth was then removed, and the staining was compared with the ISO gray scale, to determine the color transfer.

A white cotton rubbing cloth was used, and moisture pick-up was 100% of its dry weight by moistening it with distilled water and squeezing it. The same approach as in the dry rubbing process was used. The results of wet rubbing were judged by the ISO gray scale for staining.

## 3.4 Light fastness test

A Light Fastness Tester (Mercury-Tungsten Lamp M237) was used following the ISO 105 B02 standard method. The light fastness test measures the fading resistance of a dyed material to a particular light source, usually a sun-like light source. This test is used to assess the compatibility of a dye with the fabric being dyed and its resistance to light to ensure that the product will perform well and meet the standard in practical use. Dyed cotton fabric samples; prepared at different concentrations of dye (0.5%, 1%, 2% and 4%,) were evaluated. Calibration and cleaning of the test chamber. Samples were exposed to light for a fixed period (usually hours or days depending on the test requirements) along with the Blue Wool Standards. After exposure, the fading of the test samples was compared with standard lighting conditions. Light fastness was measured on a scale from 1 to 8, with 1 for very poor and 8 for excellent.

### 3.5 Color fastness to washing

This test aims to determine the resistance of the color of a dyed fabric to washing at 60°C, thereby simulating domestic or commercial laundry conditions. The resistance of the dye to the fabric and whether the dye will transfer or fade to adjacent fabrics when washed. The test was performed on a Launderometer, a device that is commonly used in a laboratory for ISO 105 C03 testing. This apparatus simulates the washing parameters such as time, temperature, and agitation to ensure repeatable results. For dye transfer evaluation (cotton or wool/polyester blend). Phosphate-free detergent was used in the presence of steel balls for mechanical agitation, according to the ISO standard. A 10 cm x 4 cm piece of this dyed fabric was cut and stitched with a piece of undyed adjacent fabric of 10 cm x 4 cm. Standard detergent solution of 4 g/L in deionized water was prepared. The liquor ratio was kept at 50:1 (50 mL of solution to 1 g of fabric). The test vessel was placed in the Launderometer and the timer was set for 30 minutes. The machine was run to simulate 60°C washing. Color change of the dyed fabric was evaluated using the Gray Scale for Color Change. The degree of staining on adjacent fabric was measured in terms of gray scale for staining.

### 3.6 Color evaluation using a spectrophotometer

The color characteristics of the dyed fabric samples under controlled conditions were evaluated using a spectrophotometer. The measurement of color deviation, color uniformity, and color fastness are provided, which gives a statistical measurement for textile dyeing, ensuring reproducibility and quality control in dyeing through this process. Colorimetric data ( $L^*$ ,  $a^*$ ,  $b^*$ ,  $C^*$ ,  $h^*$ , and K/S) of dyed cotton fabric was measured in terms of CIELAB values using a reflectance spectrophotometer. Here,  $a^*$  (+ve values) represents the degree of redness,  $a^*$  (-ve values) expresses the greenness shade of the fabric while positive  $b^*$  values show a degree of yellowness and negative  $b^*$  values represent the blueness shade of the dyed fabric while  $h^*$  is hue angle ( $0^\circ$ - $360^\circ$ ) and  $C^*$  is chroma and  $L^*$  is lightness (ranging from 0-100 where 0 is for black and 100 is for white fabric) using Eq. 2.2. K/S value of dyed fabric was calculated using Kubelka–Munk Eq. 2.1.

$K/S = (1 - R^2)/2R$ ..... 3.1

Where R is percentage reflectance, K and S are coefficients of absorbance and scattering.

$$\Delta E^* = (\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2 \dots\dots\dots 3.2$$

## 4. Results and discussion

### 4.1 Synthesis of novel reactive dyes

The successful synthesis of the novel reactive dyes was confirmed through the sequential completion of diazotization, azo coupling, and subsequent nucleophilic substitution reactions involving cyanuric chloride and choline chloride. The adopted synthetic route was designed to produce reactive dyes possessing both excellent affinity towards cotton cellulose and enhanced functional properties, including improved ultraviolet protection and environmentally friendly salt-free dyeing behavior. The overall synthesis strategy followed the methodology presented in this section and consisted of three principal steps: (i) preparation of the diazonium salt, (ii) formation of the azo chromophore through coupling with the synthesized coupling components (O-ester and P-ester), and (iii) introduction of the monochlorotriazine reactive system followed by modification with choline chloride to obtain the final reactive dye.

The first step in the procedure involved the preparation of the aromatic amine through the diazotization process in acid solution by the addition of sodium nitrite at a temperature range of 0–5°C. It was important to keep this low temperature because it ensured stability of the diazonium ion intermediate and prevented its decomposition. The diazonium ion formed was immediately coupled with the prepared coupling compound in mild alkali solution to form the azo intermediate. Coloration was observed during the coupling process, showing the presence of the azo (-N = N-) chromophore. Formation of coloration is typical of an azo dye because of the extensive  $\pi$ -electron delocalization.

Once the azo-coupling reaction was completed, cyanuric chloride was employed as the reactive link for the chromophore and the cellulose fiber. Cyanuric chloride was chosen because of the three chlorines that exist in the triazine ring, each with different levels of reactivity, thus allowing substitution to occur in a stepwise manner at increasing levels of temperature. The first chlorine atom was replaced by the azo derivative at a low temperature to retain the triazine ring structure, whereas replacement with the second chlorine resulted in choline chloride as a hydrophilic group. The addition of the choline chloride was hoped to increase the solubility of the dye molecules in water and enhance the exhaustion of the dyes during dyeing processes, besides reducing the need for inorganic electrolytes during dyeing of cotton.

All through the process of synthesis, care was taken to ensure that all reaction parameters such as temperature, pH, and reaction times were optimized to enhance product formation and reduce any side reactions. The dyes formed from this reaction had a unique color, implying that the desired dye molecules had been produced. Even though there was no direct measurement of yield or purity of the synthesized dyes through the isolation or chromatographic techniques in this research, the successful dyeing of cotton textiles, along with FTIR characterization and textile performance tests, imply that the synthesized dyes contained the desired functional groups.

After synthesis of the dyes, they were used for dyeing of cotton fabric in different concentrations (0.5%, 1%, 2%, and 4% o.w.f.) in order to examine the effect of the dye concentration on the intensity of coloration, UV shielding capacity, antimicrobial activity, and color fastness properties. This is because the selected concentrations would enable the evaluation of the newly developed dyes within the range of dyeing and enable direct comparison of their properties at varying dye concentrations. The performance of the dyes has been discussed in the subsequent sections to demonstrate the dyeing efficiency.

As can be seen from the results obtained, the developed synthetic method allowed for the synthesis of reactive dyes possessing such structural groups such as azo chromophore, monochlorotriazine reactive group, and choline chloride functionality. In the context of this work, the purpose of using such a combination of structural elements in the dye structure was to improve its substantivity, promote covalent fixation with cellulose fibers, and provide better functional properties without the use of excessive amounts of salt during dyeing.

## 4.2 Proposed reaction mechanism

The preparation of these reactive dyes through synthesis entailed several sequential organic reactions, which included diazotization, azo coupling, nucleophilic substitution of cyanuric chloride, and modification using choline chloride. These reactions were aimed at incorporating different structures within the reactive dye molecules such that, at the end of the reactions, the multifunctional reactive dyes were formed and had the capability of bonding with cellulose molecules. The overall reaction pathway is illustrated in Fig. 4.2, in which the individual reaction mechanisms are presented in step wise.

## 4.3 FTIR characterization

The FTIR spectra of the synthesized ortho and para position in reactive dyes were comparatively analyzed to verify the characteristic functional groups present in both structures and to evaluate the effect of ester position on their vibrational behavior. Both spectra are shown in Fig. 4.3; the major absorption features expected for the proposed azo-based reactive dye structure, indicating that the principal functional groups were retained in both positional derivatives. The broad absorption observed in the  $3400\text{--}3450\text{ cm}^{-1}$  region is attributed mainly to  $\text{--OH}$  stretching, consistent with the hydroxyl containing aromatic component of the dye molecule [38]. The absorption in the region between  $1600\text{--}1650\text{ cm}^{-1}$  is linked with the conjugated aromatic system and may involve the contribution from azo ( $\text{--N=N--}$ ) linkage. The FTIR also reported the same characteristics for hetero-functional azo reactive dyes, where these regions are used as supporting evidence for the presence of the conjugated chromophoric framework [36].

The most relevant differences between the ortho and para structures are observed in the fingerprint region, particularly between approximately  $1500$  and  $1000\text{ cm}^{-1}$ . The absorptions in this region can be attributed to overlapping  $\text{C--O}$ ,  $\text{C--N}$ , aromatic deformation, and sulfonate related vibrations together with contributions from the substituted triazine reactive group. The presence of these bands is consistent with the multifunctional nature of the synthesized dye, in which the aromatic chromophore, sulfonated

component and triazine-based reactive functionality occur within the same molecular structure. It has been established that cyanuric chloride can go through successive nucleophilic substitution reaction, thus making it extensively used in preparing triazine groups into reactive dyes [39].

The effect of position on the ester group can be related to the fingerprint region in particular. The ester group in the dye, which is positioned ortho to another substituent, can be closer to that substituent and may thus experience a different kind of steric interaction and electronic environment. Such proximity may influence the vibration of the ester C = O and C–O groups and consequently modify the intensity or position of bands in the fingerprint region. In contrast, the para-position dye has greater spatial separation between the substituents, producing a comparatively different steric and electronic environment. Therefore, the observed differences between the two spectra are interpreted as changes in the vibrational environment associated with the positional arrangement of the ester-containing aromatic component rather than as evidence of fundamentally different functional groups. Structural differences in reactive dyes have similarly been associated with changes in their molecular characteristics and subsequent textile performance [10].

## 4.4 UV-vis analysis

The UV–Visible spectra of the synthesized ortho- and para-position reactive dyes exhibited characteristic absorption bands in the ultraviolet and visible regions, confirming the presence of aromatic and extended conjugated chromophoric systems. The absorption bands observed in the lower wavelength region, particularly around 225 and 280–290 nm, can be attributed mainly to  $\pi \rightarrow \pi$  electronic transitions associated with the aromatic and conjugated structural units of the synthesized dyes. The presence of several aromatic rings and conjugated groups results in overlapping absorption bands illustrated in Fig. 4.4; which is typical of complex azo dye structures [37].

The most important difference between the two positional dyes was observed in the visible region. The ortho-position dye showed an illustrative  $\lambda_{\text{max}}$  of approximately 405 nm, whereas the para-position dye showed  $\lambda_{\text{max}}$  at approximately 415 nm. The shift of approximately 10 nm toward a longer wavelength in the para derivative represents a bathochromic (red) shift. A red shift indicates absorption at a longer wavelength and therefore a lower-energy electronic gtransition, according to the relationship ( $E = hc/\lambda$ ). In comparison, the shorter-wavelength absorption of the ortho derivative can be described as a relative hypsochromic (blue) shift with respect to the para derivative [40].

The difference in  $\lambda_{\text{max}}$  can be related to the different steric and electronic environments produced by the ortho and para positioning of the ester-containing aromatic component. In the ortho structure, the closer proximity of the ester group to the neighboring substituent may produce greater steric interaction and influence the orientation of the adjacent aromatic units. This can affect the effective conjugation and electron distribution of the azo chromophore. For the para structure, increased distance between the substituent groups may result in an entirely different structure, which would allow better delocalization in the conjugated system. Consequently, the para derivative absorbs a slightly longer wavelength than the

ortho derivative. Such changes in substitution pattern and molecular structure are known to influence the electronic absorption behavior of azo dyes [41].

The common absorption features observed for both dyes indicate that the basic conjugated azo-reactive dye framework was retained in both positional derivatives, while the difference in  $\lambda_{\text{max}}$  demonstrates that ester positioning modifies the electronic environment of the chromophore. The approximately 10 nm red shift observed for the para derivative therefore provides useful comparative evidence of the influence of positional isomerism on the optical properties of the synthesized dyes. However,  $\lambda_{\text{max}}$  alone should not be considered definitive proof of the exact ortho or para structure; rather, it provides complementary evidence alongside the FTIR and structural information obtained from the synthesis.

## 4.5 Ultraviolet protection factor (UPF)

The UV-protection results demonstrate in Fig. 4.5 (a-c), a remarkable improvement in the ultraviolet protective performance of the dyed fabrics compared with the unmodified fabric. The untreated fabric showed approximately 58% UVA and 57% UVB protection, with an overall UPF of about 7, indicating comparatively low inherent UV protection. After application of the synthesized reactive dyes, UVA and UVB protection increased to above 90% for both ortho- and para-position dyes. This improvement can be attributed to the presence of the extended aromatic-azo conjugated system of the synthesized dyes, which can absorb ultraviolet radiation and consequently reduce its transmission through the textile. The ability of dyes to enhance UV protection through absorption of UV radiation by their chromophoric structures has also been reported for dyed textile materials [42].

A clear concentration-dependent improvement was observed. For the ortho dye, UVA protection increased from approximately 93% at 0.5% concentration to 96% at 4%, while UVB protection increased from approximately 92% to 98%. Similarly, the para dye provided approximately 92% UVA and 93% UVB protection at 0.5%, increasing to approximately 97% and 98%, respectively, at 4%. The corresponding UPF increased substantially from about 7 for the untreated fabric to approximately 19–22 at 0.5% and 38–40 at 4% dye concentration. The higher protection at 4% can reasonably be associated with the greater quantity of dye molecules present within/on the cotton substrate, providing a larger number of chromophoric sites capable of absorbing incident UV radiation. Increased dye concentration and darker/deeper coloration are commonly associated with enhanced UV protection in textile fabrics because greater absorption reduces UV transmission [43].

Comparison of the positional derivatives shows that the para dye provided slightly higher overall UPF values than the ortho dye, particularly at the corresponding concentrations. The UPF was approximately 21–22 for para 0.5% compared with 19–20 for ortho 0.5%, while at 4% the values were approximately 39–40 and 38–39, respectively. This small difference may be related to the different molecular arrangement produced by the ortho and para positioning of the ester-containing aromatic component. The para-arrangement may provide a comparatively favorable electronic environment for conjugated chromophore, allowing efficient absorption of UV radiation. However, because the difference between

the two positional dyes is relatively small, the results indicate that dye concentration has a more pronounced effect on UV protection than positional variation [44]. The substantial increase from UPF 7 in the untreated fabric to nearly 40 in the 4% dyed fabrics is therefore the principal finding.

Overall, the results confirm that incorporation of the synthesized reactive dyes substantially improved the UV-A, UV-B, and overall UPF performance of the cotton fabric. Both positional derivatives provided more than 90% protection against UV-A and UV-B at the investigated concentrations, while the highest dye concentrations showed the highest UV protection. The 4% para-treated fabric showed the highest UPF ( $\approx 40$ ), followed closely by the 4% ortho-treated fabric ( $\approx 39$ ). These findings demonstrate that the synthesized reactive dyes can provide an additional functional benefit as UV-protective textile colorants, besides their primary role in coloration. The observed enhancement is consistent with established research showing that textile construction, dye structure, dye concentration, and colour depth can strongly influence UV transmission and UPF of fabrics [45].

## 4.6 Light fastness evaluation

The light fastness results of the synthesized reactive dyes indicate that both ortho- and para-position dyes provided moderate resistance to light-induced shade change, although their performance varied with dye concentration. For the ortho-position dye, the light fastness ratings were approximately 4.0, 4.5, 4.0, and 3.5 at 0.5, 1, 2 and 4% concentrations, respectively presented in Fig. 4.6. The highest rating of 4.5 was obtained at 1% concentration, indicating comparatively excellent resistance to shade change at this concentration. At concentrations of 2% and 4%, the rating slightly fell to 4.0 and 3.5, respectively, implying that raising the dye concentration from 1% would not have improved the stability of light. [46].

For the para-position dye, the corresponding ratings were approximately 4.0, 3.5, 3.5, and 2.5 at 0.5, 1, 2 and 4% concentrations. The 0.5% solution exhibited excellent light fastness ( $\approx 4.0$ ), but when the concentration was increased, there was a gradual reduction in the resistance to light. The lowest rating of approximately 2.5 was observed at 4% concentration, indicating comparatively greater shade change after light exposure. Thus, the para derivative showed a more pronounced concentration-dependent decline in light fastness than the ortho derivative.

Comparison of the two positional derivatives shows that the ortho dye generally exhibited better light fastness than the para dye, particularly at higher concentrations. At 0.5%, both dyes showed a similar rating of approximately 4.0; however, at 1% the ortho dye reached 4.5 compared with 3.5 for the para dye. At 2%, the ortho dye maintained a rating of about 4.0, while the para dye remained at 3.5. The difference became more evident at 4%, where the ortho dye showed a rating of approximately 3.5 compared with 2.5 for the para dye. This difference may be related to the different molecular environments produced by the ortho and para positioning of the ester-containing aromatic component, which can influence molecular conformation, electronic delocalization and the stability of the azo chromophore under light exposure. Structural features and molecular interactions are recognized as important factors governing the light fastness of azo dyes [47].

The slight reduction in light fastness at higher concentrations may also be associated with increased dye aggregation within the fibre. At higher dye concentrations, greater numbers of dye molecules are present in close proximity, which can modify their molecular environment and influence photochemical degradation processes [48]. Overall, the results demonstrate that dye concentration and positional arrangement both influence light fastness. The ortho dye at 1% concentration exhibited the best performance ( $\approx 4.5$ ), whereas the para dye at 4% showed the lowest rating ( $\approx 2.5$ ). Therefore, the ortho-position dye appears comparatively more suitable where improved resistance to light-induced shade change is required, while excessive concentration of the para derivative may adversely affect its light-fastness performance.

## 4.7 Rubbing fastness

The rubbing fastness results of the synthesized reactive dyed fabrics are presented in Fig. 3.6 for both dry and wet rubbing conditions. Samples in Fig. 4.7 (a–d) represent the ortho-position dye applied at 0.5, 1, 2 and 4% concentrations, respectively, while Fig. 4.7 (e–h) represents the para-position dye at the same concentrations. The results show excellent dry rubbing fastness, with all dyed samples achieving a rating of approximately 4.5. This indicates a very low transfer of colour from the dyed fabric to the adjacent rubbing material under dry conditions. The consistently high ratings across all concentrations demonstrate that dye concentration and positional arrangement had little effect on dry colour transfer, suggesting effective fixation of both synthesized reactive dyes onto the cotton substrate.

Under wet rubbing conditions, slightly lower ratings were observed compared with dry rubbing. The ortho samples a–d showed a rating of approximately 4.0 at all concentrations, whereas the para samples e and f (0.5 and 1%) showed the highest rating of approximately 4.5, followed by g and h (2 and 4%), which showed approximately 4.0. The minor decline in wet rubbing fastness is expected due to the fact that water can cause swelling of fibres, hence facilitating the detachment or migration of little dye from the fabric in its non-fixed form. However, a rating of 4.0–4.5 denotes good to excellent wet colour fastness, which means that most of the dye fixed to cotton fibres was successful.

Comparison between the two types of derivatives revealed that ortho dye possessed constant rubbing fastness properties, giving a result of about 4.5 in dry state and 4.0 in wet state for all tested concentrations. Para dye possessed excellent dry rubbing fastness (about 4.5) while its wet rubbing fastness was slightly higher (about 4.5) at 0.5 and 1% and reduced to about 4.0 at 2 and 4%. Slight difference between ortho and para derivatives indicates that the dye position has little effect on the rubbing fastness while the high values can be attributed to the fixation of dye. This is because reactive dyes can establish covalent bonds with cellulose and hence contribute to good resistance to rubbing and wet rubbing [31].

Overall, the rubbing fastness properties of the synthesized reactive dyes were very satisfactory with dry ratings of about 4.5 for all samples and wet ratings of 4.0 to 4.5. In view of the lack of any considerable drop in ratings despite increased dye concentrations, it may be concluded that increased concentrations

of dyes do not have an adverse impact on rubbing fastness. Thus, it may be concluded that both ortho and para reactive dyes were efficiently bonded with the cotton fabric to yield dyeing with very good rubbing fastness properties. High values of rubbing fastness are highly favorable for practical purposes because of good durability of shades thus obtained.

## 4.8 Washing fastness

The washing fastness results of the synthesized reactive dye-treated fabrics are presented in Fig. 4.8, where samples a–d represent the ortho-position dye applied at 0.5, 1, 2 and 4% concentrations, respectively, while samples e–h represent the para-position dye at the corresponding concentrations. The washing fastness was evaluated in terms of change in shade, staining of cotton, and staining of nylon. Overall, the synthesized dyes exhibited good to excellent washing fastness, indicating effective fixation of the dyes onto the cotton substrate and good resistance to removal during washing.

For change in shade, the ortho dye samples showed ratings of approximately 4.0, 4.5, 4.0, and 3.5 at 0.5, 1, 2 and 4%, respectively. The para dye showed ratings of approximately 4.0, 3.5, 3.5, and 2.5 at the same concentrations. In this case, the ortho dye showed better shade change resistance compared to the para dye, especially in high concentrations. The best rating (4.5) was attained by the ortho dye when the concentration was 1%, while the worst rating (2.5) was achieved by the para dye in a concentration of 4%. The decrease in the shade change resistance rating in higher concentrations could have been caused by the large amount of dye in the fiber. However, the majority of the samples maintained a rating of 3.5 or higher [49].

These staining results also confirm the satisfactory fixation of the synthesized dyes. In case of cotton staining, the ortho dyes have a staining rating of about 4.0, 3.5, 3.5, and 3.5, whereas the para dyes have a staining rating of about 4.0, 3.5, 3.5, and 3.0, respectively. The slight decline in the staining rating at higher concentrations, especially 4% para dye, reflects the slight migration of the color to the adjacent cotton during washing. But even then, the staining rating falls under the good fastness category. On the other hand, in case of staining on nylon, the rating was about 4.5 in all the samples. This shows the very low migration of the synthesized dyes on the adjacent nylon fabric [50].

The overall washing-fastness behavior can be attributed mainly to the reactive nature of the synthesized dyes. Reactive dyes are designed to form strong, preferably covalent bonds with hydroxyl groups of cellulose under suitable dyeing conditions. Such fiber–dye fixation reduces the tendency of the colorant to migrate from the cotton during subsequent washing. The consistently high nylon-staining ratings and generally good cotton-staining ratings support effective fixation of both ortho- and para-position dyes. The positional difference produced some variation in shade change and staining, but the overall performance of both derivatives remained satisfactory. Similar evaluation of color change and staining is carried out using standardized washing-fastness procedures such as ISO 105-C06, in which color change of the dyed material and staining of adjacent fabrics are assessed using standardized grey scales [51].

## 4.9 Antibacterial activity

The antibacterial activity of the synthesized reactive dye-treated fabric was evaluated against *Staphylococcus aureus* using the agar diffusion method. The fabric samples treated with 0.5% and 4% dye concentrations did not produce any distinct or measurable zone of inhibition around the specimens. Bacterial growth remained visible in the immediate vicinity of both treated fabrics, indicating that the synthesized reactive dye did not exhibit detectable antibacterial activity against *S. aureus* under the applied experimental conditions shown in Fig. 4.9.

The 4% concentration, despite containing a higher amount of dye than the 0.5% treatment, also failed to produce a visible inhibition zone. Therefore, there was no observable effect on antibacterial properties as the dye concentration increased from 0.5% to 4%. This clearly indicates that the prepared dye is mostly used as a reactive dye and does not give any antimicrobial property to the cotton cloth. However, the reference sample showed the presence of an inhibition zone, which means the bacterial growth was active. The absence of a comparable zone around the dyed fabrics therefore supports the conclusion that the lack of inhibition was associated with the synthesized dye-treated samples rather than failure of the test conditions.

The absence of antibacterial activity may be related to the strong fixation of the reactive dye within the cotton fibre and the lack of sufficiently accessible antimicrobial groups at the fabric surface. Since agar diffusion depends on the migration of an active antimicrobial component from the fabric into the surrounding agar, a strong fibre-bound reactive dye may not diffuse sufficiently to inhibit bacterial growth. Therefore, the present findings should be reported specifically as “no detectable antibacterial activity against *S. aureus* by the agar diffusion method” rather than claiming complete absence of antimicrobial potential.

## 5. Conclusions

The present study successfully developed and evaluated novel azo-based reactive dyes containing a monochlorotriazine reactive group and choline-derived functionality for salt-free dyeing of 100% cotton fabric. The synthesized ortho and para-position dyes were tested at 0.5-4% o.w.f. without the use of inorganic salts. Their ability to provide good coloration and fixation of cotton fabric at the chosen alkaline conditions was confirmed. The data provided by FTIR and UV visible spectroscopy confirmed the proposed structures and conjugated chromophoric system. There was an evident improvement in the UV protection of the dyed materials with the UPF going up from about 7 to almost 40. In addition, the two dyes proved themselves to be good to excellent rubbers and washers. However, ortho dyes were more resistant to light fading and shade shifting than their para counterparts. The latter offered somewhat better protection against UV radiation. No antibacterial activity was detected because of the agar diffusion test using the bacteria of *S. aureus*. In general, the results obtained show the possibility to use the prepared dyes as reactive colorants with UV protective properties for cotton fabrics.

## Declarations

## Author Contribution

Abdur Rehman has designed the project and supervised it. Mr. Ayyaz has conducted all the research work along with testing, while Dr. Kashif Iqbal has helped in results interpretation and writing.

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## Figures

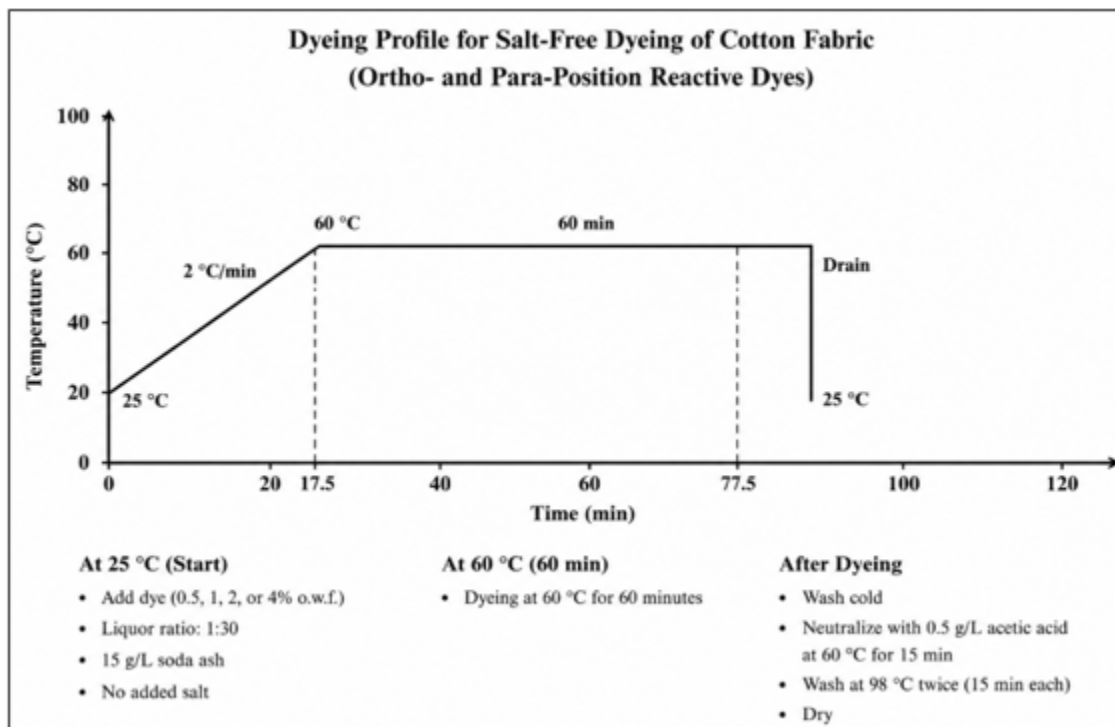
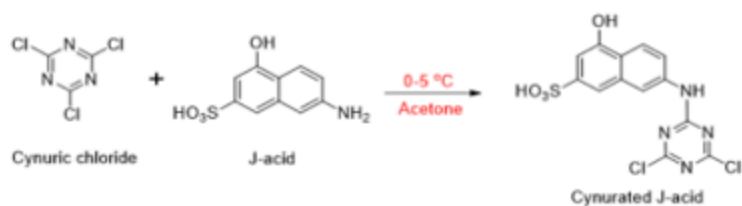


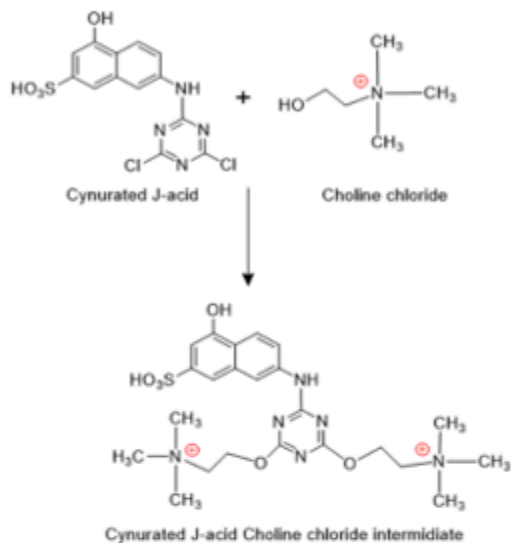
**Figure 1**

Figure 2.1: Dyeing profile of application of synthesized reactive dye

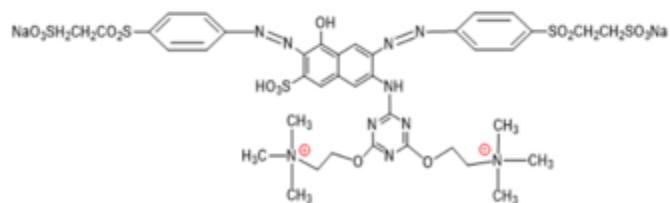
### Step 1



### Step 2



### Step 3 (a) Reaction at para position



### Step 3 (b) Reaction at ortho position

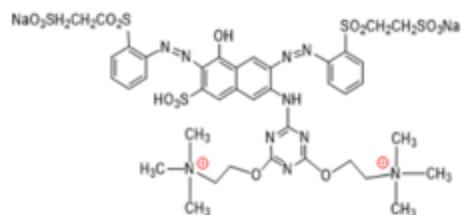


Figure 2

Figure 4.2: Proposed mechanism of prepared reactive dyes ortho and para ester positions

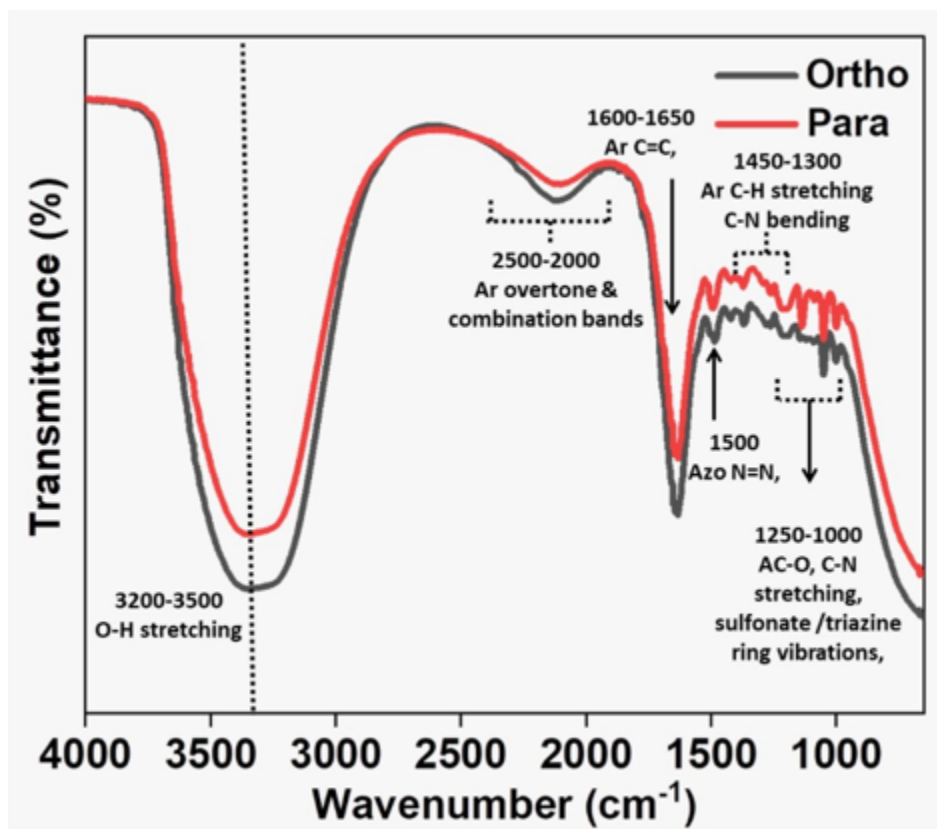


Figure 3

Figure 4.3: FTIR spectra of ortho and para ester of synthesized reactive dye

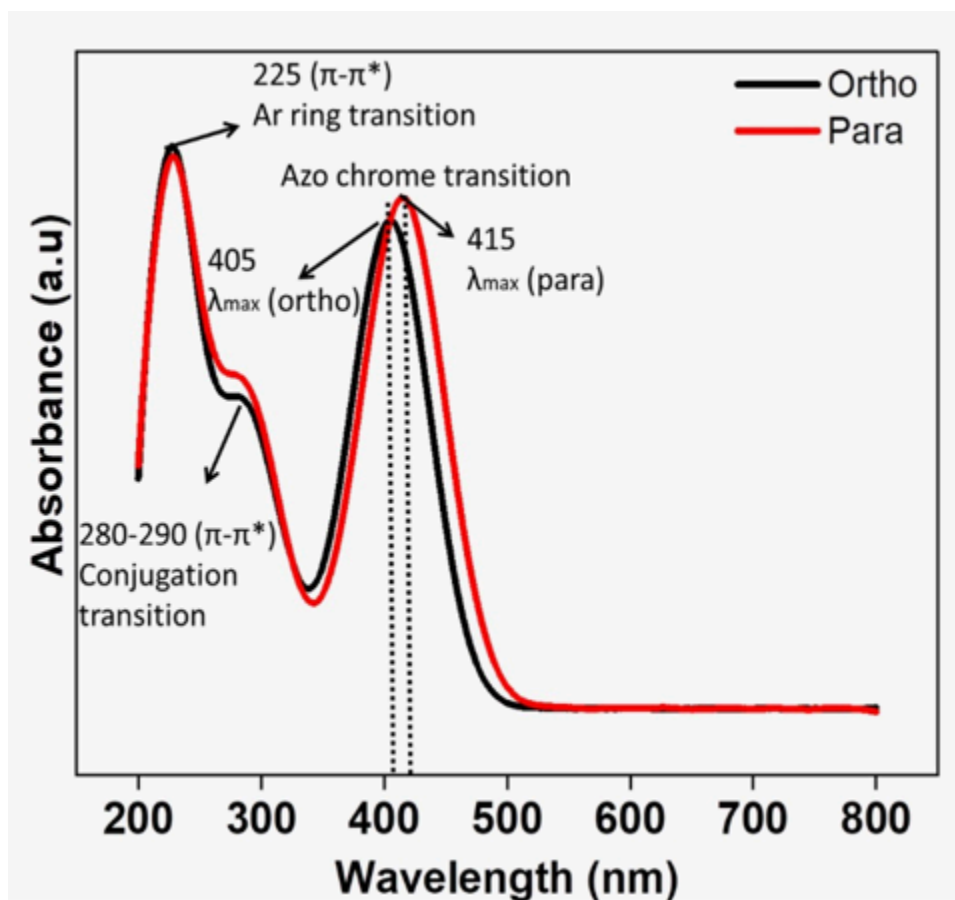


Figure 4

Figure 4.4: UV-vis spectra of ortho and para ester of synthesized reactive dye

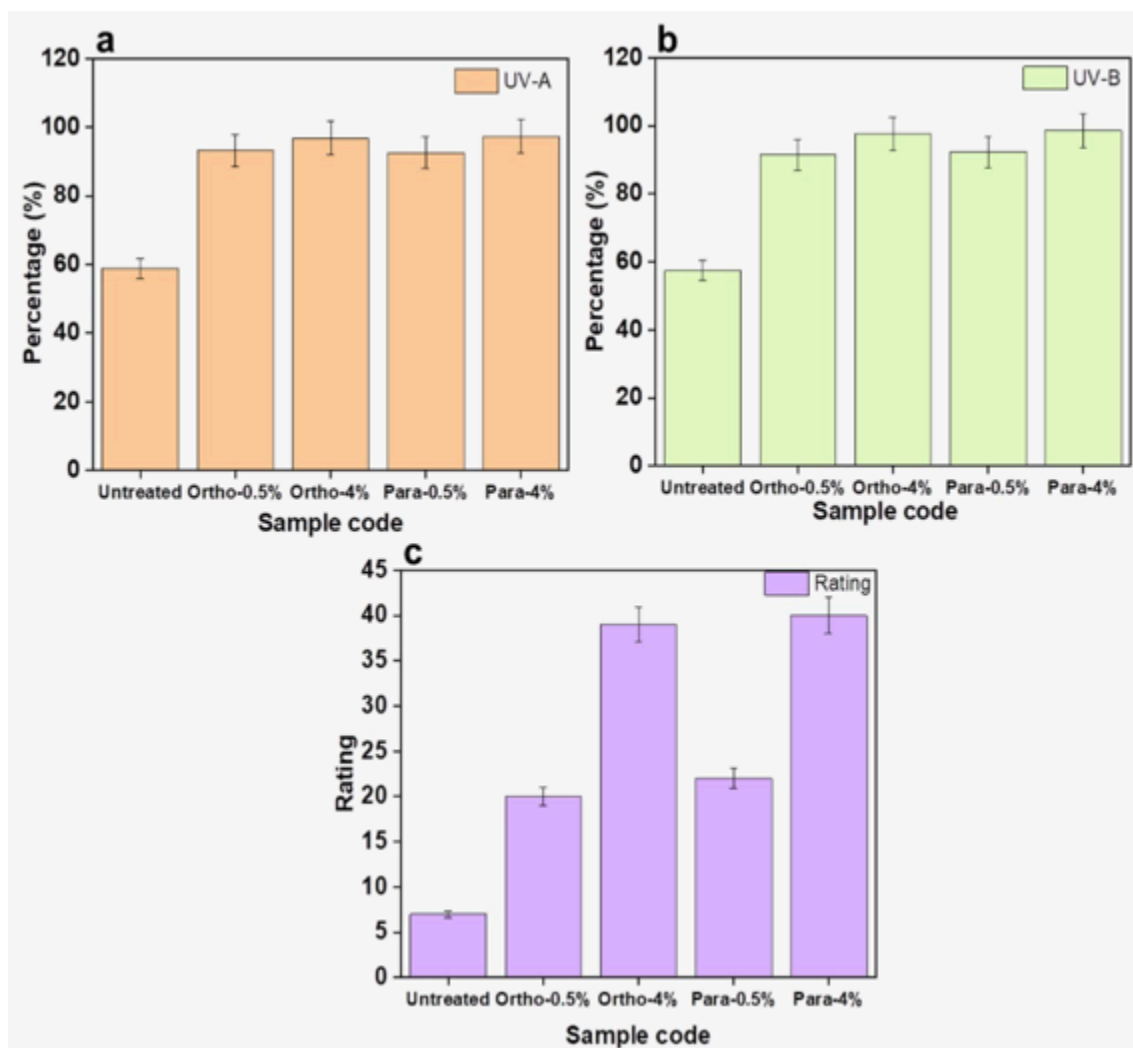


Figure 5

Figure 4.5: (a) UV-A percentage, (b) UV-B percentage and (c) UPF rating of untreated, ortho, para dyed fabric (0.5 and 4 %)

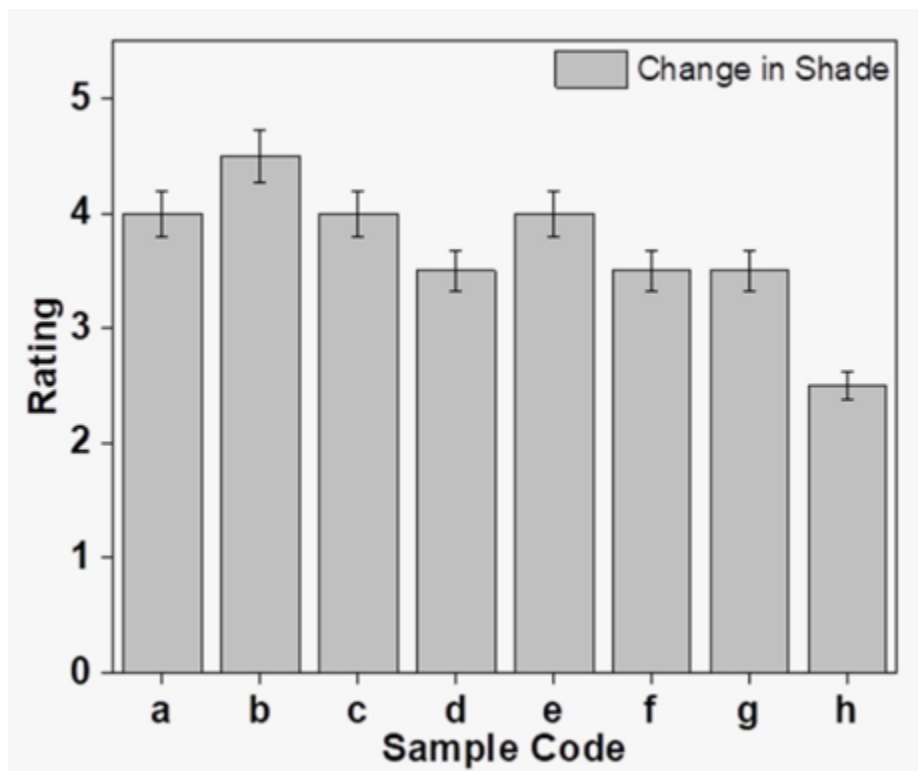


Figure 6

Figure 4.6: Lightfastness of the prepared reactive dyes ortho (a-d) and para (e-h) ester position

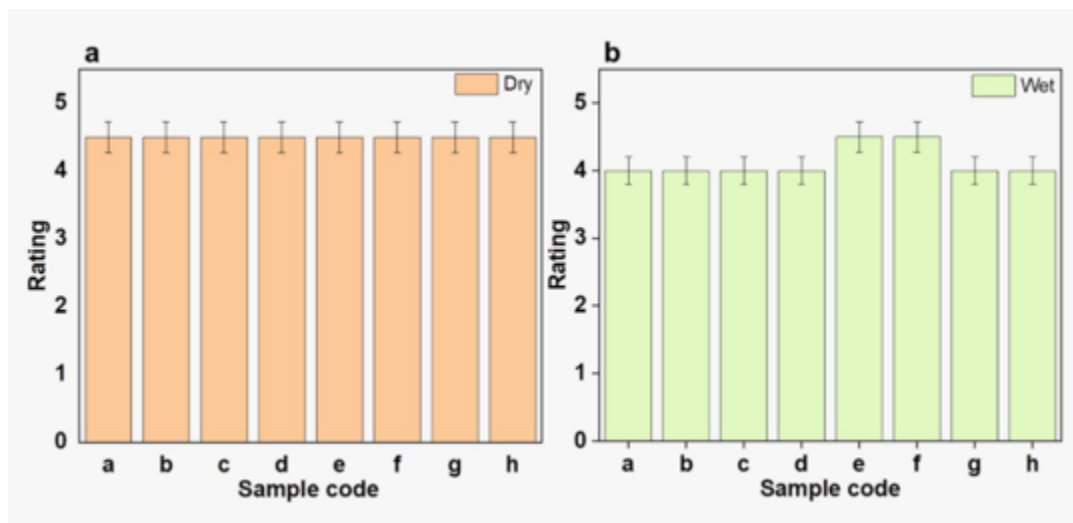


Figure 7

Figure 4.7: Dry and wet rubbing fastness of the dyes dyed fabric with ortho (a-d) and para (e-h) ester position

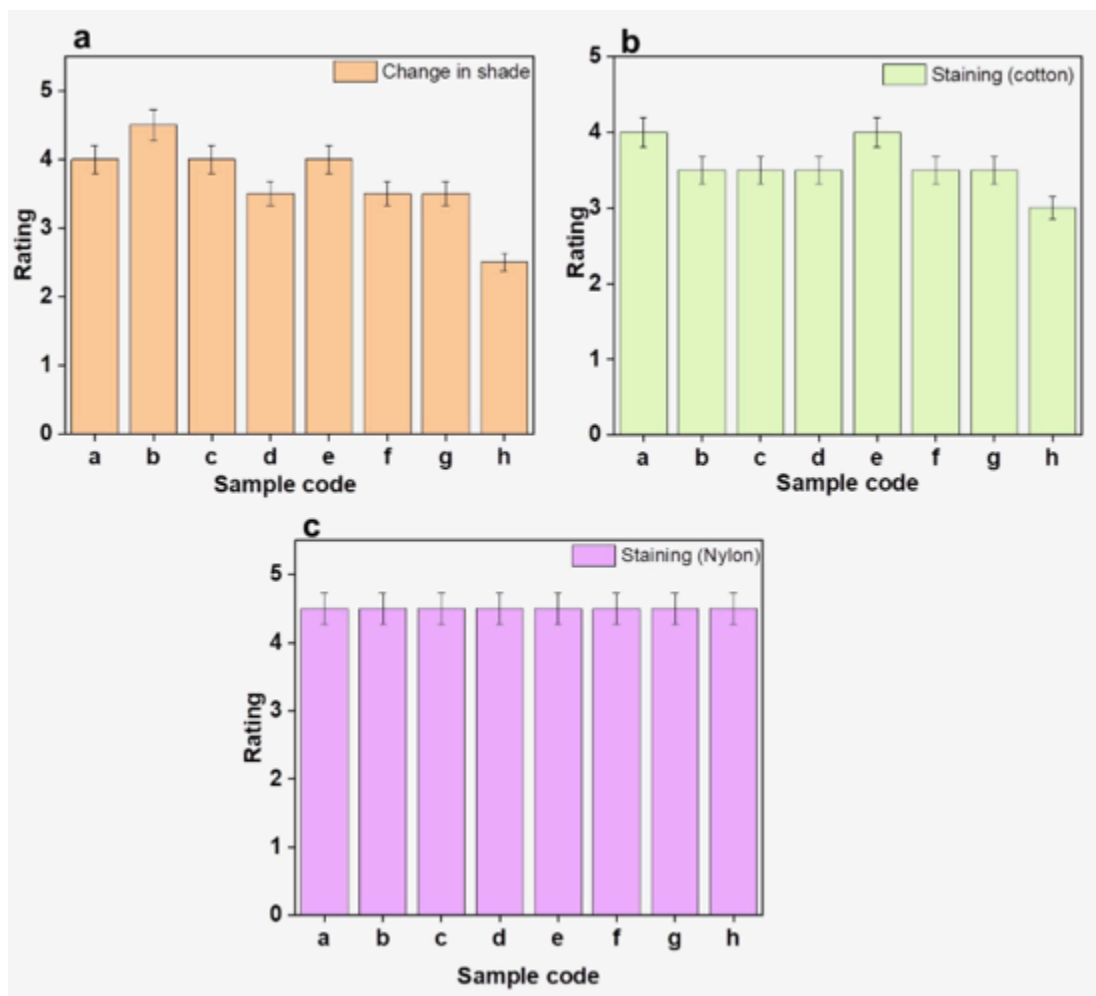


Figure 8

Figure 4.8: Washing fastness of the dyed fabric including (a) change in shade, (b) cotton staining and (c) nylon staining with ortho (a-d) and para (e-h) ester position

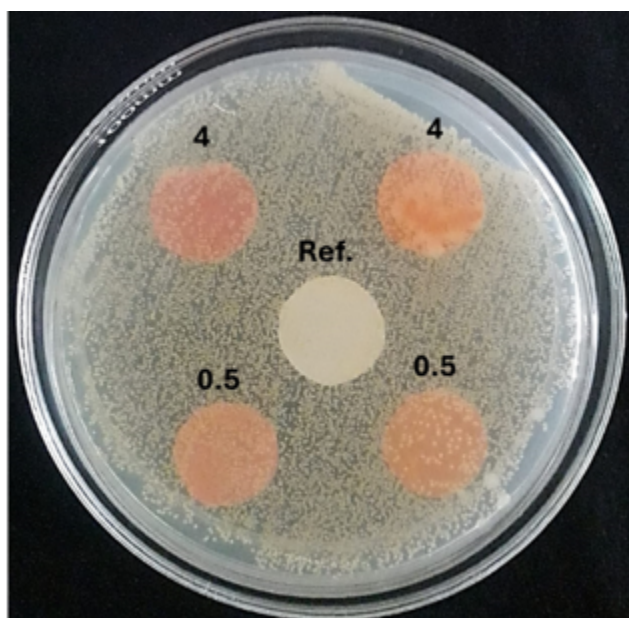


**Figure 9**

Figure 4.9: Antibacterial activity qualitative against *S. aureus* gram positive of untreated reference sample and treated with ortho and para (0.5 and 4 %)