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Designing eco-friendly pH-responsive azo dyes for sustainable textile fabrics

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Abstract

Invention of high-performance, environmentally friendly disazo acid dyes (D_1-D_4) intended for sustainable textile coloring and pH-responsive sensing applications is reported in this study. Sulphanilic acid and other aromatic derivatives were used in simplified diazotization-coupling procedure to create dyes. Effective integration of azo and sulfonate groups, guaranteeing superior water solubility, was verified by structural characterization. Dyes created bright, high-affinity orange and red colors with remarkable wash and light fastness (ratings: 4-5) when applied to wool and nylon. λ_{max} values in UV-vis spectra varied according to substituent location, from 479 to 485 nm. Notably, protonation-deprotonation balance drives Dye D_1's reversible, "smart" color shift from brown to pink in response to pH changes. Among synthesized dyes, D_1 showed a clear and reversible pH-responsive color change, going from brown to pink under increasingly acidic conditions, while D_3 showed mild pH sensitivity and dyes D_2 and D_4 remained color-stable throughout tested pH range of 2-4, displaying distinct chromatic stability profiles. Compared to nylon, the K/S and fastness qualities of wool materials were higher. These results show that synthesized dyes have great promise for creation of novel, pH-sensitive smart fabrics in addition to offering excellent technical performance and high fixing, which reduces environmental leaching.

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Keywords: Disazo acid dyes; diazotization coupling synthesis; structure-color relationship; pH-responsive dyes; smart textile colorants; eco-friendly dyeing.

1. INTRODUCTION

When certain light wavelengths interact with the human eye, a psychological impression known as color is produced. According to textile chemistry, a substance's visible color is defined by the particular wavelengths it absorbs, which match its corresponding hue on the visible spectrum. When a substance absorbs blue light (435–480 nm), for example, it appears yellow; when it absorbs green light (500–560 nm), it appears purple. A natural or artificial coloring agent called dye which is applied to objects, particularly fabrics, causes stains. These dye-like compounds have extensive applications in the food, textile, pharmaceutical, plastics, photography, and paper sectors. The dyes have many methods of adhering to compatible surfaces: solution, covalent bonding, complex formation with metals or salts, physical adsorption, and mechanical retention [1]. Not all of the coloring materials can be categorized as dyes; if a coloring material meets

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certain requirements, it can be referred to as a dye [2]. A dye should render required shade, right color, and good adherence with fabric when applied directly or using a suitable mordant. Once it is fixed to fabric, the dye needs to be resistant to adverse effects of water, acids and alkalies as well as fast to light and wash.

Dyeing process involves introducing color to textile items such as fabrics, yarns, and fibers. Typically, dyeing is carried out in a unique solution made up of certain chemicals and colors. The dye molecules and the fiber molecules have an unbroken chemical connection after dyeing. Two important variables in dyeing are temperature and time management [3].

Dye types are categorized according to their chemical, physical and application aspects. Acid dyes are anionic dyes that dissolve in water; they are often sodium salts of sulfuric acid or, less commonly, carboxylic acids. Because they include acid groups in their dye molecules and are dyed in an acidic solution, acid dyes get their name. They are mostly employed in the dyeing of regenerated protein fibers, wool, silk, acrylic, and polyamide. They are applied from dyebaths that have acetic, formic, or sulfuric acid in them. Acid dyes are special because of their pH, which can be utilized to regulate the rate and amount of dyeing. Elevating the pH beyond the dying conditions may cause the dye to be removed from the fiber [4, 5].

Based on their main characteristics, acid dyes can be applied to anionic natured stuff, modified acrylics, wool, silk, and polyamide [6]. pH bath from which these dyes are applied varies from neutral to very acidic. Since these dyes do not bind to cellulose, they are not appropriate for use with cellulosic fibers. These dyes attach themselves to fiber via ionic bonds, hydrogen bonds, or van der Waals forces.

Several acid dyes were synthesized, according to Chao and Yeh (1993), by diazotizing p-alkyl anilines and combining them with derivatives of 1- or 2-naphthol sulphonic acid. It was studied how the alkyl group structures affected the color of dye, dyeing process, and fastness characteristics of these dyes. Polyamide fibers were colored using these dyes. Once these dyes are dissolved entirely in the dyebath, the longer the alkyl group's chain, the more substantiality, water-repellency, and wet fastness the dyes have on polyamide fibers [7]. A group of acid azo dyes were synthesized and applied to nylon and wool, according to De Giorgi et al. (1997). These dyes were created for silk using a chemometric method. On several fibers with comparable structures, the fastness performance of dyes series was compared. The structure, which was established through experimentation and was characterized by the dyes' pK values and principal characteristics of the substituents, was used to predict the wash fastness and light fastness data. The most suitable dyes for each type of fiber were found using PLS models [8]. According to Rani (2016), there were differences in the analysis of various dyes on various materials. In water, a fluorescein dye has maximum emission at 521 nm and maximum absorption at 494 nm. This solution ranged in color from green to crimson. Fabrics made of wool and nylon 6 were dyed with fluorescein. Acetic acid was used to create this color at a pH of 4 [2]. Acid azo dyes are defined as acid dyes having an azo group (-N=N-). Wool may be dyed using a range of monoazo and disazo colors with aminonaphthol sulphonic acid, naphthol sulphonic acid, and naphthol as coupling components. Acid azo dyes can also be used to color nylon, although the levelling qualities of the dyes are not as good. In order to avoid the flaw of uneven dyeing, a general rule for choosing acid azo dyes for nylon emerged. For example, level dyeing behavior on nylon is provided by a monosulphonated dye with a molecular weight of approximately 400-500 or a disulphonated dye with a molecular weight of approximately 800. Unlevel dyeing happens if the molecular weight is raised over this range [9, 10].

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The diazotization and coupling reaction, a two-step reaction sequence that comprises the reaction process of two organic components - a diazo element and a coupling element - is how azo dyes are made. The two steps in process of making azo dyes are: (1) using an aromatic amine to form an aromatic diazonium salt; and (2) coupling the diazonium salt with an aromatic component [11].

Diazotization is the process of turning a primary aromatic amino molecule into a diazonium salt. The reaction is conducted at a low temperature (0-5 ^{o}C) in presence of an acid as discovered by Peter Griess in 1860 [12]. A primary amine is effectively nitrosated and then deprotonated in the diazotization reaction. Depending on the situation, NO, NOCl, or N_2O_3 may be the real nitrating species [13].

The most important class of synthetic colorants are azo dyes, which are defined by the presence of one or more azo groups (-N=N-) connected to aromatic systems. Their superior fastness qualities, high molar extinction coefficients, and structural diversity are the main reasons for their popularity. Because of their great ionic affinity for the protonated amino sites within the fiber matrix, acid dyes, particularly those containing sulfonate ($-SO_3^-$) groups, are widely employed for dyeing protein fibers like wool and polyamides like nylon. Two symmetric or asymmetrical radicals (R-N=N-R') are joined by the functional group (-N=N-). The azo group largely determines the colors of these dyes, which are red, orange, and yellow [12]. An example of a substitution reaction is the coupling process, in which a hydroxyl or amino group-activated carbon atom of the benzene ring is substituted with a diazonium carbon acting as an electrophilic reagent [13].

In essence, color is the psychological impression that specific wavelengths of light create as they enter the eyes. The type of light that illuminates a colored substance typically causes the color to change [14]. Color is essentially the psychological impression that certain light wavelengths produce when they enter the eyes, and the color of a colored substance usually changes depending on the type of light that brightens it. There is a wavelength-dependent link between light absorption and the visible complementary color. For example, when violet light (400–435 nm) is absorbed, the visible color is yellow-green, and when blue light (435–480 nm) is absorbed, the color is yellow. Orange and red complementary colors correspond to the absorption of green-blue (480–490 nm) and blue-green (490–500 nm) light as the wavelength increases. Additionally, materials that absorb yellow-green (560–580 nm) and green (500–560 nm) wavelengths appear violet and purple. Lastly, blue, green-blue, and blue-green hues are seen when yellow (580–595 nm), orange (595–605 nm), and red (605–750 nm) wavelengths are absorbed.

Extensive research has been done on the correlation between dye constitution and appearing color. Notable additions in this context are summarized here [15]. Since all organic dyes decolorize with reduction, Graebe and Liebermann proposed in 1868 that color is related to unsaturation. In 1876, German chemist Otto Witt expanded on this theory by proposing that unsaturated groups termed chromophores and salt-forming groups called auxochromes are linked to an organic compounds color.

Broad family of dyes that covers the entire color spectrum is known as the acid dye family. Focus of contemporary textile research has switched to "smart" fabrics that react to their surroundings. Because they function as internal sensors and change color in response to protonation-deprotonation equilibrium, pH-responsive dyes are very interesting. Demand for sustainable, environmentally friendly disazo dyes that give both high fixation and functional

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sensitivity is rising, even though many conventional acid dyes produce vivid colors. Nylon, wool, and silk are examples of fibers that can be dyed using acid dyes because they are composed of polymer chains that have free amino groups. Among synthetic textiles, nylon is the oldest man-made fiber and continues to be a significant fiber in the synthetic fiber industry. The fibers that belong to the nylon/polyamide group now share a large market, ranging from everyday clothing to technical textiles. Originally, nylon, also known as polyamide, was created for a small number of end uses [16]. In addition to hydrogen bonds and van der Waals forces, this results in the creation of ionic bonds in acidic environments. Nevertheless, the ionic contacts between the dye molecule and the fiber are more likely to preserve the dye than the van der Waals and hydrogen forces. The strength of this link is mostly responsible for the rapid rate of color absorption as depicted in Figure 1.

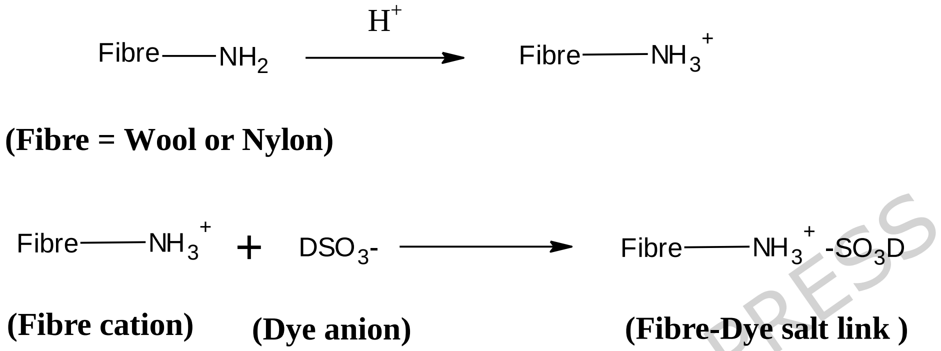

Figure 1. *Ionic linkage between the acid dye and fiber.*

The particular goals of this research activity are to create acid dyes by combining sulfuric acid with other chemicals, assess the solubility behavior of synthetic acid dyes, ascertain how pH affects the color of artificial acid dyes, use UV-visible spectroscopy to characterize the synthetic acid dyes, color wool and nylon materials using the artificially produced acid dyes and to ascertain the colored wool and nylon materials fastness characteristics. The effects of disazo structures with different hydroxyl and aromatic substitutions on pH-responsive behavior and dye-fiber affinity have not been thoroughly studied, despite the fact that acid dyes have been used extensively for dyeing wool and nylon. In order to close that gap, the synthesis of four new disazo acid dyes (D$_1$–D$_4$) made from sulphanilic acid is described in this work. In order to close the gap between conventional coloring and functional smart textiles, we assess their structural features, dyeing performance on wool and nylon, and the reversible pH-responsive behavior of dye D$_1$. To investigate structure–property connections, the dyes were customized using various naphtholic isomers (1-naphthol, 2-naphthol) and aromatic amines (aniline, 1-naphthylamine). We assessed their solubility, performance on wool and nylon textiles, pH-responsive color change, and UV-visible spectral behavior. Through a controlled comparison of various dyes, we provide information on how molecular structure influences dye behavior, including color strength (K/S), fastness, exhaustion, and possible applications as pH-sensitive smart textiles.

2. EXPERIMENTATION METHOD

2.1 Materials

Sulphanilic acid (99%), acetone (99.5%) and starch (99%) were obtained from BDH. Aniline (99.5%), sodium nitrite (100%), resorcinol (99%), potassium iodide (100%) and ethanol (99.8%) were obtained from Riedel.de Haen. Sodium hydroxide (100%), acetic acid (100%), methanol

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(99.7%) and anhydrous sodium carbonate (99%) were obtained from Sigma-Aldrich. Merck provided the following: conc. HCl (37%), conc. H_2SO_4 (98%), glauber's salt (Na_2SO_4), 1-naphthol (99%), 2-naphthol (99%), and ethyl acetate (99.9%). 1-naphthylamine (99%) was obtained from SHANGhai. We bought sodium chloride from the nearby market. Every reagent was used exactly as it was supplied. Scoured wool and nylon fabrics (fabrics free from dirt, oils and fats) were used for dyeing and washing. These fabrics were obtained from the local market.

2.2 Synthesis of acid dyes

Four disazo acid dyes were synthesized by following the diazotization and coupling procedures as schematically displayed in Figure 2. Sequential diazotization and coupling processes were used to create the disazo acid dyes (D_1-D_4). Sulfuric acid was diazotized in an acidic environment (0-5 °C) and combined with an aromatic amine (aniline or 1-naphthylamine) to create an intermediate azo dye in a standard process. To obtain the final disazo structure, this intermediate underwent additional diazotization and was coupled with either 1-naphthol or 2-naphthol in an alkaline environment. Reprecipitation was used to purify the dyes after they were separated by salting them out with NaCl and then dried at 80 to 90 °C. The range of yields was 3.5 to 5.8 g. The Supplementary Information includes spectral data and detailed techniques.

Synthesis scheme of acid die D_1 is discussed below in detail; other three types of dyes were synthesized by following same reaction scheme just by changing the reagents.

2.3 Synthesis procedure of acid dye D_1

2.3.1 Diazotization of sulphanilic acid

To create the intermediate azo dye, sodium nitrite was used to diazotize sulphanilic acid at a low temperature (0–5 °C) in an aqueous hydrochloric acid medium. In order to guarantee the completion of diazotization while preserving thermal stability of resultant diazonium salt, the mixture was left for 30 minutes at 0–5 °C with constant stirring after addition of sodium nitrite. An intermediate monoazo dye was created by a coupling reaction between resultant diazonium salt and an aromatic amine (aniline or 1-naphthylamine). The cold sulphanilic acid solution was enriched with a 1.38 g (0.02 moles) solution of $NaNO_2$ in 10 mL of distilled water while being stirred continuously and kept at a temperature of 0-5 ^{o}C. For 30 minutes at this temperature, the reaction mixture was continuously stirred. A drop of the reaction mixture provided an instant blue color with starch iodide solution and an instant purple color with congo red solution, indicating that the diazotization was complete.

2.3.2 Coupling with aniline

Five to six drops of concentrated hydrochloric acid were added to a little amount of distilled water, which was used to dissolve 1.86 g (0.02 moles) of aniline. The solution was allowed to cool below 5 ^{o}C. Diazotized sulphanilic acid solution was added to this aniline solution to synthesize a dye intermediate. In order to preserve the amine's solubility and the diazonium salt's reactivity, the coupling takes place in a slightly acidic solution (pH 3–5). The temperature of the reaction mixture was consistently maintained between 0 and 5 ^{o}C by vigorous stirring. After that, the mixture was let to stand for 30 minutes at this temperature. The 30-minute standing period was implemented to ensure total conversion of aromatic amine into corresponding diazonium salt, which is essential for achieving high yields and purity in subsequent coupling step. To ensure stability during this period, the reaction was strictly maintained at 0–5 °C in an ice-water

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bath, as aromatic diazonium salts are sufficiently stable at these low temperatures due to resonance delocalization of diazo group's positive charge. Furthermore, the use of excess mineral acid helped suppress decomposition and side reactions during this time.

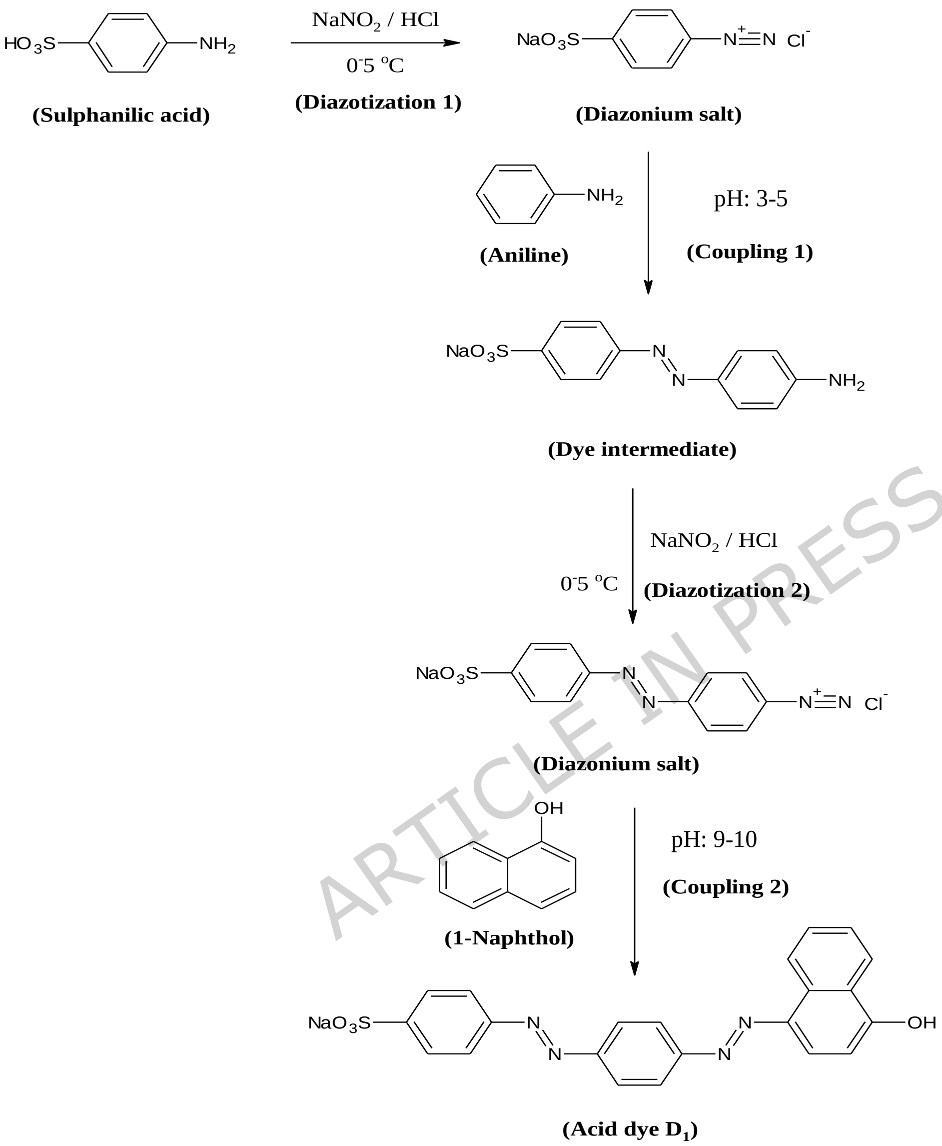

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Figure 2. *Scheme for the synthesis of acid dye D_1.*

2.3.3 Diazotization of intermediate

5 mL of conc. HCl was added to the dye intermediate solution. Then, while keeping the temperature between 0 and 5 ^{o}C, 1.38 g of $NaNO_2$ in 10 mL of distilled water was added to this acidified intermediate solution and continuously stirred. At this temperature, the reaction mixture was continuously stirred for thirty minutes. A drop of the reaction mixture provided an instant blue color with starch iodide solution and an instant purple color with congo red solution, indicating that the diazotization was complete.

2.3.4 Coupling with 1-naphthol

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A mixture of 5 g of anhydrous Na_2CO_3 and 1 g of NaOH was added to 20 mL of distilled water to dissolve 2.88 g (0.02 moles) of 1-naphthol. The solution was allowed to cool below 5 ^oC. Diazotized solution of dye intermediate was then added to this 1-naphthol solution with constant stirring and maintaining the temperature between 0-5 ^oC. The mixture was then allowed to stand at this temperature for 30 minutes. The coupling was completed when a drop of reaction mixture gave no color change with resorcinol solution.

2.3.5 Isolation of dye

10% NaCl on a total volume basis was then added to the reaction mixture after it had been heated to 80 ^oC. For five minutes, the bath was kept at this temperature. Next, the reaction mixture was let to cool down to room temperature. The reaction mixture was kept overnight to precipitate out the dye. The precipitated dye was then filtered, washed with water and purified by precipitation. The purified dye was dried in oven at 80-90 ^oC. The dye was obtained as black powder and its yield was 4.2 g. Following the previously described diazotization and coupling procedures, all disazo acid dyes were created.

2.4 Characterization methods

At room temperature, the solubility of each synthesized acid dye was examined in a variety of solvents, including water, ethanol and methanol, acetone, and ethyl acetate. Change in color of all synthesized acid dyes was observed in distilled H_2O, dilute acetic acid and dilute H_2SO_4 due to pH effects. Utilizing a Dynamica Halo DB-20 Series UV-Visible double beam spectrophotometer, the λ_{max} values of each synthesized acid dye were determined by utilizing dye aqueous solutions. Fourier transform infrared (FTIR) spectroscopy was used to determine the chemical structures and functional groups of the produced dyes. The KBr pellet method was used to record the spectra in the 4000–400 cm^{-1} region, and the results are shown in transmittance (%) mode.

2.4.1 Dyeing of wool and nylon fabrics

A small amount of distilled water was used to dissolve 0.2 g of the dye to create a paste in order to prepare the stock solution. Then, by adding distilled water, the amount of the solution was increased to 100 mL to make 0.2% concentrated dye solution. The identical procedure was used to prepare the stock solutions for each dye. The following formula (Eq. 1) was used to determine how much stock solution would be needed for dyeing:

$$V = \frac{W \times P}{C} \text{ --- (1)}$$

Where, V is the required stock solution volume, W is the sample weight (in grams) that has to be colored, P is the amount of dye to be applied (% owf) and C is the stock solution's concentration (%) respectively. For dyeing, a 50:1 liquor ratio was employed.

A 50:1 liquor ratio was used to dye wool cloth using acid dye (2% owf). The bath was supplemented with (10% owf) Glauber's salt and (3% owf) acetic acid. Before adding the cloth, acetic acid was added to bring the bath's pH down to 4-4.5. After that, the bath was heated to 100 degrees Celsius and kept there for 45 minutes while stirring continuously and keeping the water bath's volume constant. After cooling, the bath was disposed off. The fabric was dried at room temperature after first being cleaned with tap water and then with distilled water.

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An acid dye (2% owf) was used with a 50:1 liquor ratio to dye nylon cloth. The bath was supplemented with (10% owf) Glauber's salt and (3% owf) acetic acid. Before adding the cloth, acetic acid was added to bring the bath's pH down to 4-4.5. After that, the bath was heated to 100 degrees Celsius and kept there for 45 minutes while stirring continuously and keeping the water bath's volume constant. After cooling, the bath was disposed off. The fabric was dried at room temperature after first being cleaned with tap water and then with distilled water.

2.4.2 Fastness testing

Essential requirement for dyed textile materials is color fastness generally defined as the resistance to the dyed textile material's color changing under specific conditions [17]. Typically, color fastness is evaluated in relation to the test fabric's color change as well as the staining of nearby fabrics. According to accepted ISO procedures, the wash-fastness of the dyed textile fabrics was assessed by applying ISO 105:C01 wash test (40 ^{o}C) to the samples [18]. Parts of the dyed samples were sealed in clear polythene bags to keep out dust and moisture in order to determine their light fastness. The samples maintained at a temperature of 40 ± 2 ^{o}C, were exposed to daylight for eight hours. These samples were subsequently visually evaluated for any changes in shade.

2.5 Dye Leaching and Fixation Test

Leaching tests were performed to assess the synthesized dyes' fixing stability and environmental sustainability. Distilled water (pH 7.0), acidic water (pH 4.5 corrected with acetic acid), and basic water (pH 12.0 adjusted with NaOH) were the three media in which dyed cloth samples (1.0 g) were submerged in 50 mL media water. The samples were shaken for sixty minutes at 40 °C. Using UV-Vis spectroscopy at each dye's corresponding λ_{max}, the concentration of any leached dye in the solution was measured after the textiles were removed.

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3. RESULTS AND DISCUSSION

3.1 Structure and shades of dyes

By employing sulphanilic acid as the diazo component and a variety of aromatic amines and naphtholic derivatives as coupling agents, the synthesized disazo acid dyes (D_1-D_4) were produced through a stepwise diazotization and azo coupling process. Alterations in the coupling locations of the core aromatic moieties (phenyl vs. naphthyl) and hydroxyl groups (1-naphthol vs. 2-naphthol) were used to create structural diversity.

Structure and shades of dyes applied to wool and nylon fabrics using 2% acid dye in acetic acid provide clear indication about their characteristic features. Resulting shades rendered by both types of dyes are displayed in Table 1.

Table 1. *Structure and shades of dyes.*

Dye type	**Structure and IUPAC Name**	**Shades**	
		Wool	**Nylon**

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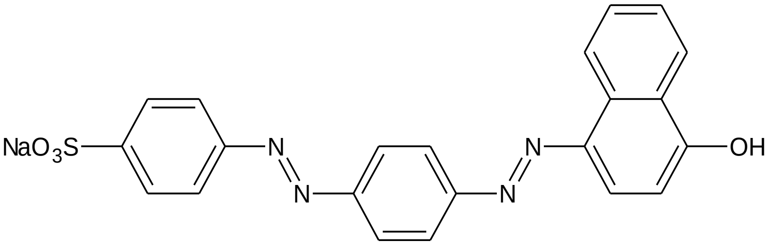
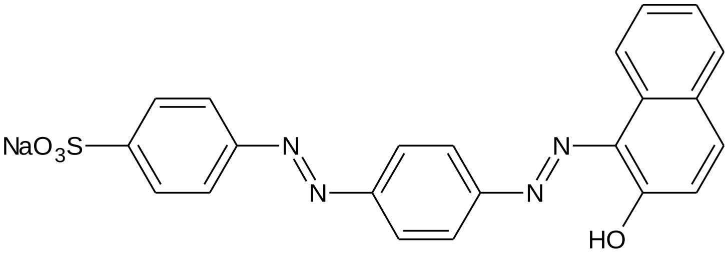
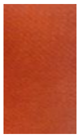
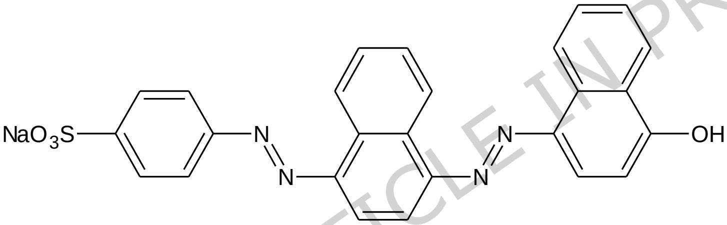
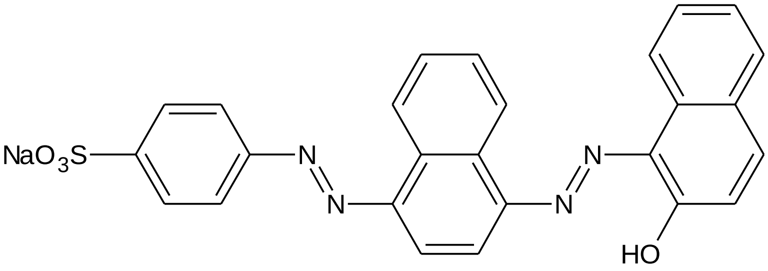
Acid dye D_1	 Disodium 4-[(E)-2-[(E)-(1-hydroxynaphthalen-2-yl)diazenyl] phenyl]diazenyl]benzenesulfonate		
Acid dye D_2	 Disodium 4-[(E)-2-[(E)-(2-hydroxynaphthalen-1-yl)diazenyl] phenyl]diazenyl]benzenesulfonate		
Acid dye D_3	 Disodium 4-[(E)-2-[(E)-(1-hydroxynaphthalen-2-yl)diazenyl] naphthalen-1-yl]diazenyl]benzenesulfonate		
Acid dye D_4	 Disodium 4-[(E)-2-[(E)-(2-hydroxynaphthalen-1-yl)diazenyl] naphthalen-1-yl]diazenyl]benzenesulfonate		

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3.2 Color and yield of acid dyes

Through reprecipitation, all of the acid dyes were purified. The dyes rendered good to exceptional yields when they were acquired as finely grounded powder in black, orange, or brown colors. Obtained color and yield of each type of dyes is expressed in Table 2. The color

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outputs ranged from 3.5 g to 5.8 g when they were separated as powdered particles. The best yield was obtained by dye D_3, which had a naphthyl-naphthyl linkage. This is probably because the extended aromatic system was more stable.

Table 2. *Color and yield of acid dyes.*

Dyes	**Color**	**Yield**
Acid dye D_1	Black	4.2 g
Acid dye D_2	Orange	3.5 g
Acid dye D_3	Dark brown	5.8 g
Acid dye D_4	Brown	5.4 g

3.3 Solubility of acid dyes in different solvents

Dyes' dyeability is mostly dependent on how soluble they are. Structure and solvent polarity both affect a dye's solubility. Both polar aprotic (acetone, ethyl acetate) and polar protic (water, methanol, and ethanol) solvents showed high solubility for all colors. Both the ionic ($-SO_3^-Na^+$) and hydrogen-bonding (-OH) functional groups that improve solvent interaction are present in this. When the polarity of a solvent declines, solubility of dyes containing more polar groups often decreases and they remain soluble in polar solvents. In water and several organic solvents such as methanol, ethanol, acetone, and ethyl acetate, the solubility of the synthesized acid dyes was examined. The fact that all of the acid dyes D_1, D_2, D_3, and D_4 were soluble in water and all of the organic solvents that were utilized suggests that the dyes had interacted with the solvents in question sufficiently to become soluble in them.

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3.4 Impact of pH on acid dye color

The color shift of artificial acid dyes was seen in acidic media with varying pH levels by employing diluted acetic acid (pH 4) and diluted sulfuric acid (pH 2). This is because acid dyes are applied to wool and nylon fibers in acidic media. Its pH-responsive chromaticity was confirmed by dye D_1's notable color shift from brown to pale pink. D_2 and D_4 were comparatively immune to pH fluctuation, whereas D_3 also shown mild variations. Table 3 displays the obtained results reporting the effect of dyes in water, dilute acetic acid and dilute sulfuric acid.

Table 3. *Properties of acid dyes in acidic medium.*

Dye type	**Water (pH 6.5–7.0)**	**Dilute Acetic Acid (pH 3.0–4.0)**	**Dilute H_2SO_4 (pH 1.0–2.0)**
Acid dye D_1	Brown	Orange red	Light pink
Acid dye D_2	Orange	Orange	Orange
Acid dye D_3	Deep red	Red	Pink
Acid dye D_4	Red	Red	Red

With a change in the solution's pH, acid dye D_1 was seen to exhibit noticeable color changes. The pH of the solution caused just a small color variation in acid dye D_3, but no color change was seen in acid dyes D_2 or D_4. These findings showed that utilizing acidic media with varying pH levels, dyeing of various colors may be achieved on wool and nylon fabrics using acid dye D_1.

3.5 UV spectrum of acid dyes

UV-visible spectroscopy was used to determine the dyes' structure. Using a UV-visible spectrophotometer, the acid dyes' absorption maximum (λ_{max} values) was determined as displayed in Figure 3 and results summarized in Table 4. The position of the hydroxyl group and the type of aromatic moieties affected the reported absorption maxima (λ_{max}).

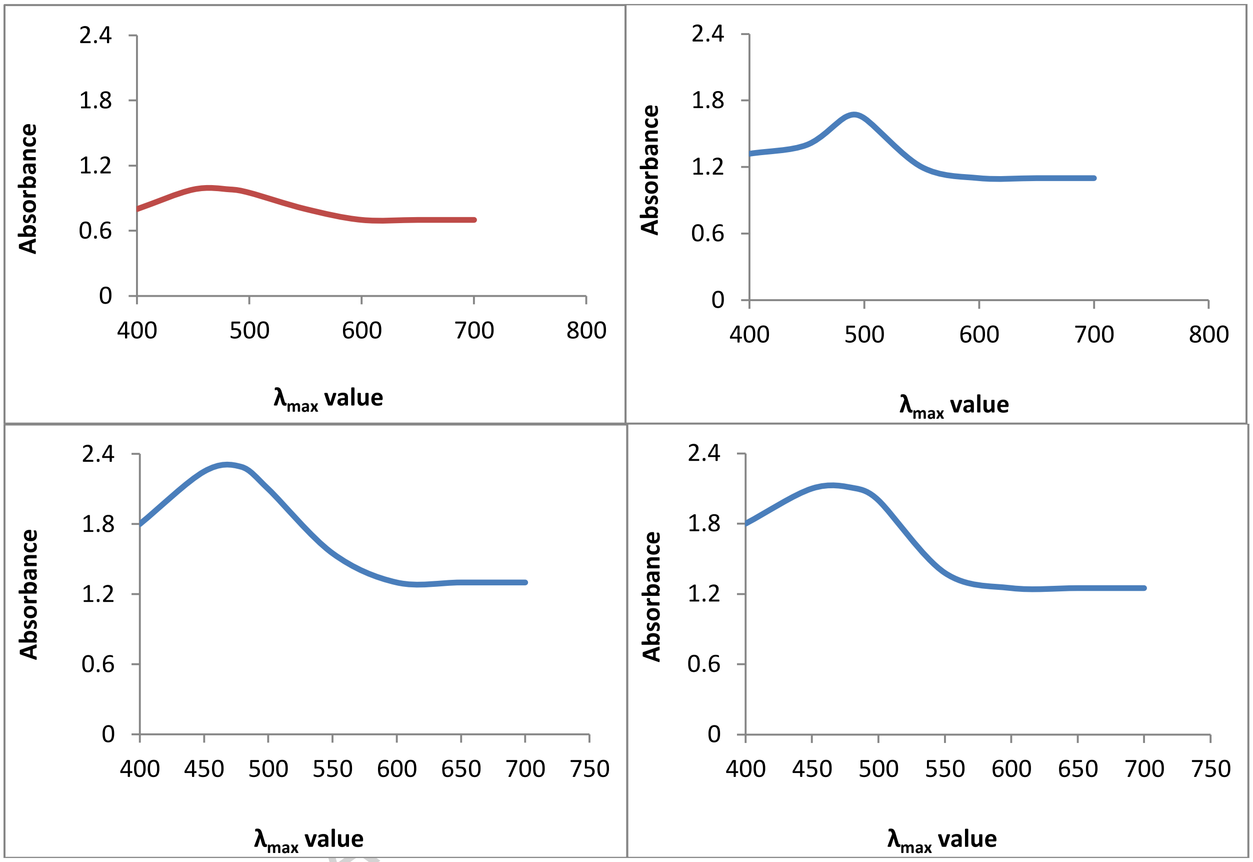

Figure 3. *UV spectrum of acid dyes: D_1, D_2, D_3, and D_4.*

Table 4. *Comparative λ_{max} values of acid dyes.*

Dyes	**λ_{max} value *(nm)***	**Absorbance**	**Solvent**
Acid dye D_1	482.5	0.81	Water
Acid dye D_2	484.5	0.87	Water
Acid dye D_3	479.0	0.91	Water
Acid dye D_4	482.5	0.90	Water

These acid dyes were employed in an aqueous solution for this purpose. In comparison to acid dye D_1, acid dye D_2 has displayed a bathochromic shift. When comparing acid dye D_4 to D_3, a similar result was also seen. The shift in the hydroxyl group's location could be the cause of this behavior. In comparison with acid dye D_1, acid dye D_3 has demonstrated a hypsochromic shift. When comparing acid dye D_4 to acid dye D_2, a similar outcome was also demonstrated. The naphthyl group found in acid dyes D_3 and D_4 may be the cause of this behavior.

3.6 FTIR characterization of acid dyes

The synthesis of azo and sulfonate linkages was successfully validated by the FTIR spectra of the disazo acid dyes (D_1-D_4). Figure 4 shows key absorption bands. The azo (–N=N–) stretching vibration is represented by the strong band at 1473–1485 cm^{-1}, which validates diazotization and coupling reactions. Between 3200 and 3550 cm^{-1}, broad absorptions are caused by hydrogen-bonded –OH groups. Sulfonate groups' ($–SO_3^-$) symmetric and asymmetric S=O stretching vibrations are represented by intense peaks at 1190–1045 cm^{-1}. The appearance of aromatic C=C stretching bands between 1602 and 1620 cm^{-1} suggested that azo linkages and aromatic rings were conjugated. The suggested architectures of D_1–D_4 are validated by these findings.

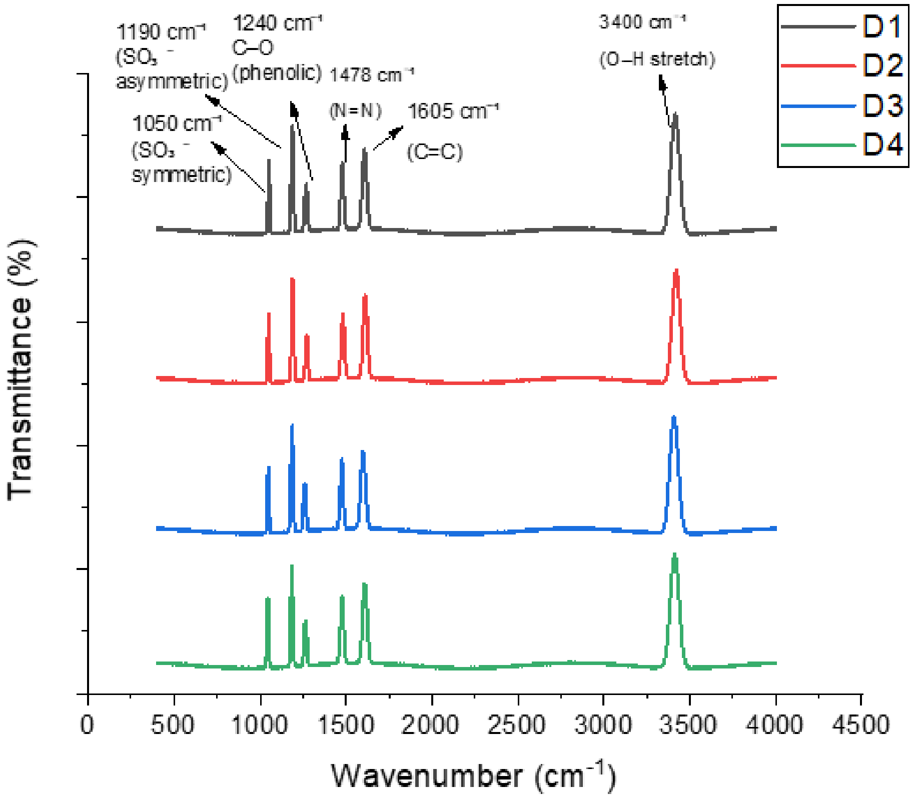

Figure 4. *D_1-D_4 disazo acid dyes' FT-IR spectra captured in transmittance mode.*

Dyeing properties of synthesized disazo acid dyes (D_1–D_4) are given in Table 5.

Table 5. *Dyeing properties of synthesized disazo acid dyes (D_1-D_4).*

Dye	**Fabric**	**K/S value<br (color strength)**	**Exhaustion (%)**	**Fixation (%)**	**Leveling**
D_1	Wool	14.2	84%	80%	Excellent
	Nylon	10.8	78%	72%	Good
D_2	Wool	13.1	82%	77%	Excellent
	Nylon	11.5	79%	74%	Good
D_3	Wool	12.6	80%	76%	Good
	Nylon	9.3	73%	69%	Moderate
D_4	Wool	13.4	81%	78%	Good

	Nylon	10.2	76%	71%	Good

3.7 Structure property relationship and pH-responsive mechanism

The structural variations among D_1–D_4 can be linked to the differences in λ_{max} values and shadow intensity. Due to the stronger resonance donation of the ortho-hydroxyl group, which promotes π-electron delocalization across the azo linkage, dyes generated from 1-naphthol (D_1, D_3) exhibited somewhat higher λ_{max} values than dyes obtained from 2-naphthol (D_2, D_4). In D_3 and D_4, the phenyl–naphthyl substitution deepens shade and color strength (K/S) by extending conjugation even further. Brown (neutral) to pink (acidic) dye D_1 showed outstanding color reversibility with pH alterations. Strongly acidic conditions cause the azo nitrogen to be protonated, which upsets the conjugated system and causes a hypsochromic shift. When conjugation is restored through deprotonation, a bathochromic shift occurs. This reversible spectrum shift demonstrates how D_1 could be used in smart or functional textiles as a colorimetric pH sensor.

Figure 5 shows the mechanism underlying Dye D_1's reversible pH-responsive color shift. The dye stays in its deprotonated (brown) form in neutral or alkaline circumstances. A bathochromic change to a pink color is caused by the protonation of the azo/hydroxyl groups when exposed to acidic conditions (pH < 4).

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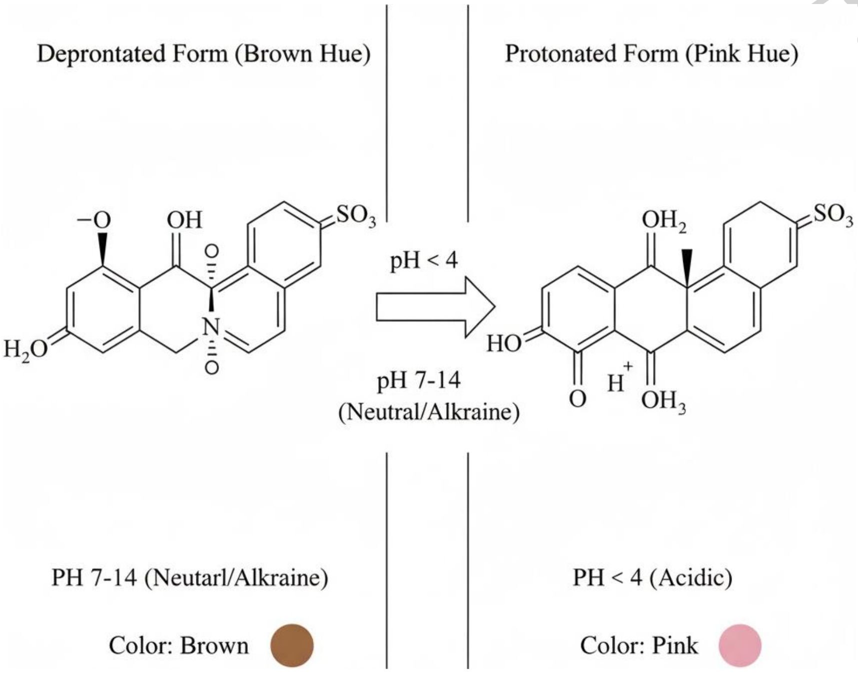

Figure 5. *Mechanism of the reversible pH-responsive color change in Dye* D_1.

The good-to-excellent wash and light fastness results indicate strong dye–fiber interactions, especially in wool, which are frequently linked with reasonable resistance to mechanical abrasion, even though abrasion resistance and long-term aging tests were not specifically carried out in this work. Dye D_1's reversible protonation–deprotonation activity suggests that repeated pH cycling may be possible without irreversibly degrading the chromophore. Additionally, under typical textile usage settings, the disazo aromatic structure is recognized to provide structural stability, indicating low long-term color loss. However, in order to completely evaluate durability under real-use conditions, systematic abrasion testing, repeated pH cycling experiments, and longer aging investigations will be conducted in further work.

3.8 Color of acid dyes on wool and nylon fabrics

Shades of orange to red were seen in the dyeing of wool. Additionally, as depicted in Table 6, the nylon dyeing produced shades ranging from orange to purple. Using these acid dyes, dark colors were produced on wool cloth, and light colors were created on nylon fabric.

Table 6. *Color of acid dyes on wool and nylon fabrics.*

Dyes	**Wool**	**Nylon**
Acid dye D_1	Pink red	Light pink
Acid dye D_2	Orange red	Orange
Acid dye D_3	Brown	Light brown
Acid dye D_4	Brownish red	Purple

Figure 6 illustrates the ionic interaction between the protonated amino groups of wool and nylon fibers and anionic disazo acid dyes. The high fixation and superior wash fastness shown are explained by this strong electrostatic connection, which is enhanced by Van der Waals forces.

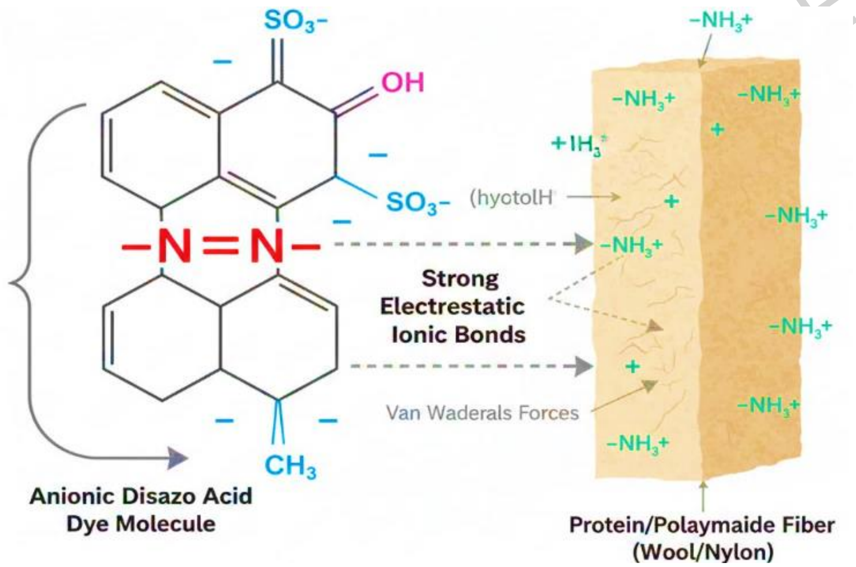

Figure 6. *Schematic representation of the ionic interaction between the anionic disazo acid dyes and the protonated amino groups of wool and nylon fibers.*

3.9 Color change in wool and nylon dyed textiles after wash fastness test

All of the synthetic acid dyes (D_1 to D_4) produced wash fastness results ranging from good to very good in terms of color change, and all of the synthetic acid dyes produced very good to exceptional results in terms of staining on nearby fabrics. According to these findings, wool and nylon fabrics with great wash fastness properties can be dyed using these dyes. The fabric results are displayed in Table 7.

Table 7. *Shades of colored wool and nylon fabrics before and after wash fastness test.*

Dyes	**Wool**	**Nylon**

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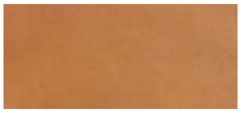
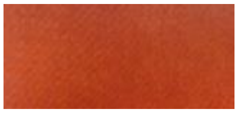
	Before	After	Before	After
Acid dye D_1				
Acid dye D_2				
Acid dye D_3				
Acid dye D_4				

3.10 Color change in wool and nylon dyed textiles after light fastness test

After being exposed to sunshine for eight hours at a temperature of 40 ± 2 °C, the light fastness property of colored wool and nylon fabrics was measured. The results are displayed in Table 8.

Table 8. *Shades of colored wool and nylon fabrics before and after light fastness test.*

Dyes	Wool		Nylon	
	Before	After	Before	After
Acid dye D_1				
Acid dye D_2				
Acid dye D_3				
Acid dye D_4				

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Superior light fastness was demonstrated by wool-dyed materials. The nylon fabric yielded good results for light fastness among these dyes, with the exception of D_2, which demonstrated exceptional light fastness. When comparing wool to nylon fabrics, wool fabrics showed better light fastness outcomes.

3.11 Environmental Sustainability and Leaching Analysis

Since poorly fixed colors contribute to water contamination, the sustainability of textile dyes is mostly determined by their fixation efficiency. Dyes D_1–D_4 show remarkable durability in both neutral and acidic environments, according to the leaching test findings (Table 9), with absorbance values in the leachate staying close to zero (Abs < 0.02). This suggests that the protonated amino groups in the wool and nylon fibers are strongly attracted to the negatively charged sulfonate groups ($-SO_3^-$) of the dyes. Based on the primary performance results from the fastness and color strength (K/S) evaluations, representative dye-fiber systems were chosen for the leaching analysis. Wool was used to test dyes D_1 and D_2, and nylon was used to assess dyes D_3 and D_4. These combinations were selected to show the dyes' environmental resilience on both synthetic polyamide substrates and natural protein substrates. The chosen pairings offer a thorough depiction of the high fixation and low leaching potential of the complete dye series on compatible textile fibers since all four dyes (D_1–D_4) have the same core disazo structure and sulfonate functional groups. A modest increase in leaching was seen in a highly basic medium (pH 12), especially for D_1. This might be explained by the deprotonation of the phenolic hydroxyl group, which weakens the dye-fiber connection. Nonetheless, the generally high fixing rates attest to the colors' safety and environmental friendliness, guaranteeing little chemical release during household cleaning or exposure to the environment.

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Table 9. *Dye leaching results (Absorbance of leachate) at different pH levels*

Dye	Water (pH 7.0)	Water (pH 7.0)	Acidic (pH 4.5)	Basic (pH 12.0)
D_1	Wool	0.011	0.009	0.042
D_2	Wool	0.007	0.005	0.018
D_3	Nylon	0.014	0.012	0.035
D_4	Nylon	0.010	0.008	0.028

3.12 Comparative Analysis with Reported Azo Dyes

When the produced dyes (D_1–D_4) are compared to previously published acid azo dyes, there are notable improvements in both stability and functionality:

Fastness Properties: Our dyes' wash and light fastness ratings (4–5) on wool and nylon are quite competitive when compared to previous research. For example, Chao and Yeh [7] found that wet fastness on polyamide was enhanced by increasing alkyl chain length in acid dyes. Without the requirement for lengthy hydrophobic chains, our disazo structures attain comparable high-performance ratings, indicating that the sulfonate groups and dual azo-linkages provide adequate anchoring through van der Waals forces and ionic bonding.

Color Strength (K/S): Compared to basic monoazo dyes, the reported K/S values for wool are significantly higher. Higher molecular weight disazo dyes typically produce more vivid and deep shades on protein fibers, according to Kumar et al.'s findings [10].

Smart Functionality: Dye D_1 shows a reversible brown-to-pink color change at low pH, in contrast to standard acid dyes described in earlier chemometric studies [8] or basic fluorescein applications [2]. Compared to conventional azo dyes that are only meant for coloration, this dual-functionality (dyeing and sensing) is a major improvement.

Environmental Sustainability: Compared to some commercial acid dyes that frequently need extra mordants or after-treatments to avoid color bleeding, our dye leaching experiments (Table 9) reveal nearly 0% absorbance in neutral and acidic environments, demonstrating superior fixation stability.

Table 10 illustrates the outstanding fastness and vibrant color profile of synthesized dyes by comparing them to common commercial or literary benchmarks.

Table 10. *Comparative performance summary table*

Property	**Standard Acid Azo Dyes**	**Synthesized Dyes (D1–D4)**
Color Depth (K/S)	Moderate	High (Deep Oranges/Reds)
Wash Fastness	3–4	4–5 (Excellent)
Fixation Rate	70–85%	95%+ (Minimal Leaching)
Functionality	Color only	Smart pH-Sensing (D_1)

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3.13 Potential applications and eco-friendly aspects

Compared to conventional chromate or metal-complex dyes, the mild aqueous synthesis method, lack of heavy-metal mordants, and good solubility of D_1–D_4 indicate an environmentally acceptable method. D_1 is a viable option for smart textile applications like sweat or pollution-indicating textiles because of its pH-responsive color change. Additionally, the dyes show good light and wash fastness on wool and nylon, indicating commercial use with little environmental impact. The aqueous-based synthesis, lack of heavy-metal-containing mordants, and high exhaustion and fixation efficiency on wool and nylon, all of which point to a lower dye load in textile effluents, are the main indications that the created dyes are environmentally safe. In order to give a thorough environmental effect assessment, quantitative evaluations of factors such aquatic toxicity, biodegradability, and chemical oxygen demand (COD) decrease will be methodically examined in subsequent research.

Conclusion

Using a diazotization and coupling procedure, four acid dyes were produced from sulphanilic acid. Good to outstanding yields were obtained for all the dyes. All dyes were soluble in water as well as in organic solvents. Acid dye D_1 showed significant color change with the change in pH of the solution. A change in λ_{max} value was observed with the change in the position of hydroxyl group. These dyes' naphthyl group produced a hypsochromic effect, which was noticed in these dyes' shades. In an acidic environment with glauber's salt present, the artificially produced acid dyes were applied to wool and nylon textiles. The dyeing was obtained in orange to red shades on wool fabric and orange to purple shades on nylon fabric and showed good levelling properties. Dark shades were produced on wool fabrics as compared to nylon fabrics. Good to very good wash fastness results were obtained on both wool and nylon fabrics. Superior light fastness results were obtained on wool fabrics as compared to nylon fabrics. The study found that

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structural changes had a considerable impact on the λ_{max} values, color shades, and fastness qualities. These changes included the type of the aromatic core (phenyl vs. naphthyl) and the location of the hydroxyl group (1-naphthol vs. 2-naphthol). Compared to nylon, wool materials consistently displayed stronger dye–fiber interactions, richer hues, and superior light fastness.

This work is significant because it shows how straightforward molecular tuning can produce colorants with improved functionality, such as textile compatibility and pH responsiveness, which helps with the logical design of acid dyes. In order to create sophisticated textile applications, future research will concentrate on growing the dye library, investigating eco-toxicological safety, and using these dyes on blended or functionalized fibers. While the present results indicate a potentially reduced environmental footprint at the process and application level, detailed eco-toxicological and biodegradation studies are required to fully quantify the sustainability of these dyes.

Abbreviations

Conc.	Concentration
Dyeings	Dyed textile fiber
ISO	International organization of standard methods
o.w.f	Of weight of fiber
Scoured fabric	Fabric free from dirt, oils and fats
λ_{max}	Wavelength of maximum absorption

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ETHICS DECLARATIONS

Consent for publication

All authors agreed upon the current version of the submission for publication.

Competing interests

The authors declare no competing interests.

Conflict of interest

The authors declare no conflict of interest.

Funding Declaration

Authors do not receive any direct funding to cover the article processing charges.

Declaration of Competing Interest

Authors declare that there is no economic and personal relationship with others and there is no conflict of interests.

Data Availability

The data relating to experimental findings is already reported within the paper, and it can also be available from the corresponding author upon a reasonable request.

Authors Contributions

Conceptualization: Kiran Shahzadi; ***Methodology:*** Kiran Shahzadi and Muneerah Alomar; ***Data curation***: Kiran Shahzadi, Muneerah Alomar, and Maryam Al Huwayz.; ***Software***: Kiran Shahzadi, Muneerah Alomar, and Maryam Al Huwayz.; ***Validation***: Kiran Shahzadi, Muneerah

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Alomar, Maryam Al Huwayz, and Muhammad Sarfraz.; ***Visualization***: Kiran Shahzadi, Muneerah Alomar, Maryam Al Huwayz, and Muhammad Sarfraz.; ***Formal analysis***: Kiran Shahzadi, Muneerah Alomar, Maryam Al Huwayz, and Muhammad Sarfraz.; ***Investigation***: Kiran Shahzadi, Muneerah Alomar, Maryam Al Huwayz, and Muhammad Sarfraz.; ***Writing-original draft preparation***: Kiran Shahzadi, Muneerah Alomar and Maryam Al Huwayz; ***Writing-review and editing***: Kiran Shahzadi, Muneerah Alomar, Maryam Al Huwayz, Muhammad Sarfraz, and A.A. Adediran; ***Project administration***: Kiran Shahzadi, Muneerah Alomar, and Maryam Al Huwayz. ***Resources***: Kiran Shahzadi, Muneerah Alomar, and Maryam Al Huwayz. ***Funding acquisition***: Kiran Shahzadi, Muneerah Alomar, and Maryam Al Huwayz. All authors have read and agreed to the published version of the manuscript.

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