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ORIGINAL RESEARCH ARTICLE

Substituent Effect on Absorption Spectra of 8-Amino-2-Hydroxyl –Napthalene- 1,6 –Disulphonic Acid-1,3,4, Thiadiazole

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ABSTRACT

Dyes are colored substances that bind chemically with fibers, becoming a part of the materials on which it is applied. Dyes are used in various fields, including printing systems, textile applications, and photoelectronic applications. Thiadiazoles are found in various structures and possess biological activity and clear importance in the pharmaceutical and industrial fields. This research centred on the substituent effect on the Absorption spectra of 8-amino-2-hydroxynaphthalene-1,6-disulphonic acid-1,3,4-thiadiazole. Four different dyes derived from the diazotization of 2-aminothiadiazole as the diazo component with R-acid, H-acid, Tobias acid and BON acid as the coupling components were synthesized. The identities of these dyes were investigated using spectroscopic techniques such as FT-IR, UV-Visible spectroscopy, and melting point measurements, with melting points ranging from 158 °C and absorption within the UV-Visible region of the electromagnetic spectrum, ranging from 360 to 480 nm. The FT-IR spectroscopy of the synthesized dyes confirms the presence of functional groups, including 3365.8 (NH/OH), 1513.3 (N=N), 1632.8 (C=N), and 1278.5 (C-S), at characteristic vibrational frequencies. The synthesized dyes yielded orange, pink, purple, and pale brown shades and exhibited very good to excellent wash fastness (4-5) and light fastness (7-8) on dyed nylon 6,6. Different substituents at various positions have been confirmed to result in shifts in the absorption maxima and changes in the intensity of absorption bands, highlighting the importance of understanding the electronics structure of the molecule for tailoring its optical properties.

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KEYWORDS
Dyes, Color and Aminothiadiazole

INTRODUCTION

Dyes are coloured substances that bind chemically to fibres, becoming part of the materials on which they are applied (Ashmawy *et al.,* 2023; Christie, 2015; Mustapha *et al.,* 2025). They are organic colour substances that, when applied to substrates, impart colour to the material by a process that at least temporarily destroys the coloured substance's crystalline structure (Brown, 1971; Magaji *et al.,* 2025). Dyes are generally applied in an aqueous solution and require a mordant to improve their fastness on fiber leather. Dyes are widely used across various fields, including printing systems, textile dyeing, and photo-electronic applications (Gregory, 1990; Asif, 2015). Nowadays, synthetic azo compounds are widely used in various applications, including medicines, cosmetics, food, paints, plastics, shipbuilding, the automotive industry, and cable manufacturing (Zollinger, 2003). However, the traditional application field of synthetic azo dyes remains the textile industry, and the finishing of fibrous materials to impart colouration and antimicrobial properties simultaneously is of great interest (Peters and Freeman, 1991; El-Apasery, 2018). This is because textile materials undergo biological degradation, and about 40% of the damage appears to be caused by microorganisms. The activities of fungi and bacteria result in reduced mechanical strength, colour changes, stains, and a stale odour (Gupta and Jain, 2012). In this regard, the use of materials with antimicrobial properties extends their service life and prevents damage from biological degradation. The manufacture of biologically active materials can be performed either by impregnation with antimicrobial compounds or by chemical reaction (by adding antimicrobial compounds via chemical bonding to the functional groups of fibre-forming polymers) Hunger, (2003). Dyes are among the compounds suitable for biocidal treatment of textile materials because some of them exhibit biological activity, resulting from the presence of antiseptic groups in their molecules that form specific interactions with the molecules of the fibrous material (Freeman and Peters, 2000). The present study focuses on the development of new eco-friendly azo dyes with good colouristic and application properties and biological activity (Helal *et al.*, 2019).

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Most conventional dyes applied to nylon 6,6 materials exhibit poor fastness to light, washing, and heat, and fall within the visible region of the electromagnetic spectrum. Hence, they exhibit insufficient bathochromic shifts compared to dyes derived from heterocyclic moieties. Moreover, most of them have low penetrability, with only a small amount of dye absorbed by nylon 6,6, resulting in significant waste in the leather industry. Therefore, synthesizing low-molecular-weight dyes derived from 2-aminothiadiazole is intended to address these problems when applied to nylon 6,6 (Koutentis, 2008; Hu *et al.*, 2014).

The use of dye in Nylon 6,6 finishing cannot be reduced because it contributes to nylon dyeing (Selivanova *et al.*, 2022; Gbadegesin *et al.*, 2025). They are considered easy to use, relatively cheap, and provide strong colour (Kamel *et al.*, 2011). Thiophene-fused heterocyclic organosulfur systems, especially the thieno moiety, have attracted significant attention because they exhibit a wide spectrum of biological activities, including antibacterial and human receptor-binding inhibition (Griffiths, 1984; Katritzky *et al.*, 2008). About 80% of all dyestuffs used in the leather industry are azo dyes; they are synthetic colours, and most of them contain only one azo group, which, when applied, provides good substantivity and better light fastness on Nylon (Kudelko *et al.*, 2020). Moreover, they also find applications in material chemistry and chemical engineering. Thus, due to their intriguing properties and applications. Therefore, it is hoped that synthesizing a low-molecular-weight mono-azo dye derived from 2-aminothiadiazole as the diazo component will yield better exhaustion and good fastness properties on Nylon 6,6. This research work aims to determine the substituent effect on the absorption spectra of 8-amino-2-hydroxy naphthalene 1, 6 disulphonic acid- 1, 3, 4-thiadiazole, by (i) Synthesis of substituent effect on absorption spectra of 8-amino-2-hydroxy-napthelene 1, 6, disulphonic acid 1,3,4 thiadiazole; (ii) Spectroscopic assessment of the synthesized dyes using UV-visible spectroscopy and FT-IR; and (iii) Application of the dyes on nylon 6-6- and substrate and fastness properties assessment.

MATERIALS AND METHODS

Materials

Chemicals

8-amino-l-naphthol-3,6-disulphonic acid (H-acid). Ferrous sulphate, Sodium acetate, Ethanol, Dimethyl sulfoxide, Methanol, Concentrated. Gamma acid, thiosemicarbazide and Sodium Nitrite; Tobias acid, Gamma acid, and naphthionic acid.

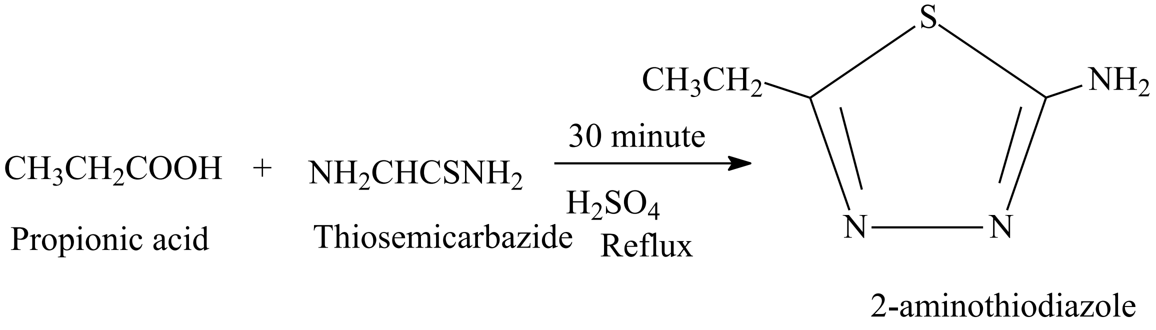
Apparatus

Ice bath, P^H meter, round bottom flask, spatula, thermometric water bath, Gallenkamp melting point apparatus, Agilent CARRY 630 FT-IR spectrophotometer, beaker 250 cm and 1cm, thermometer 0°C to 100°C, funnel, pipette, measuring cylinder, drying oven, Aluminum foil, petri dishes, weighing balance and Agilent CARRY 300 UV-visible spectroscopy.

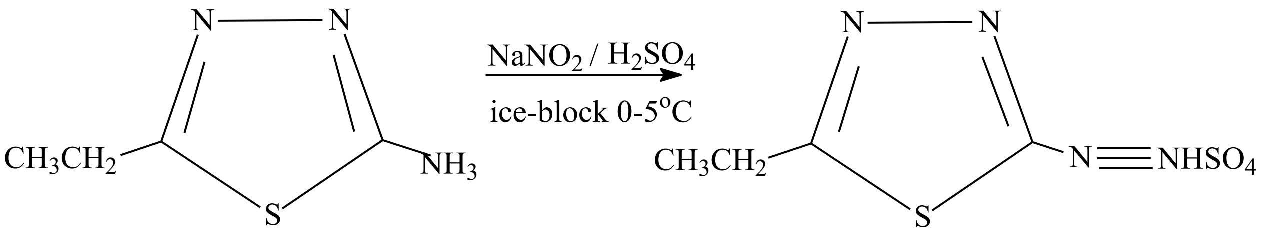
Method

Synthesis of 2-aminothiozole intermediate

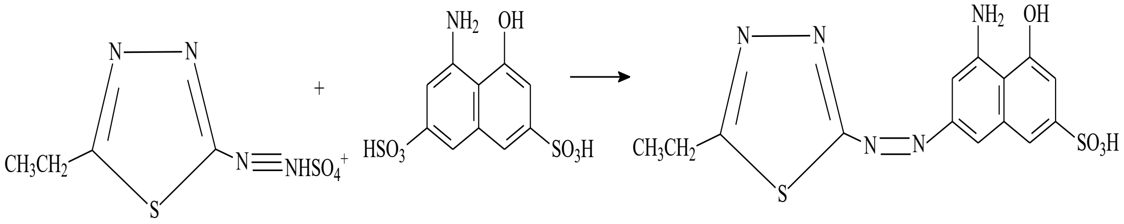
Propionic acid (0.1 mol, 6 ml) and thiosemicarbazide (0.1 mol) in concentrated sulphuric acid (2.0 ml) were refluxed gently for 30 minutes (Scheme 1). The solution was allowed to cool and was poured into a beaker containing crushed ice. The separated solid was filtered, suspended in water, and basified with sodium bicarbonate, then kept overnight. The obtained solid was filtered, washed with water, dried, and crystallized from ethanol to obtain a colourless solid (Zanoni *et al.*, 2010).



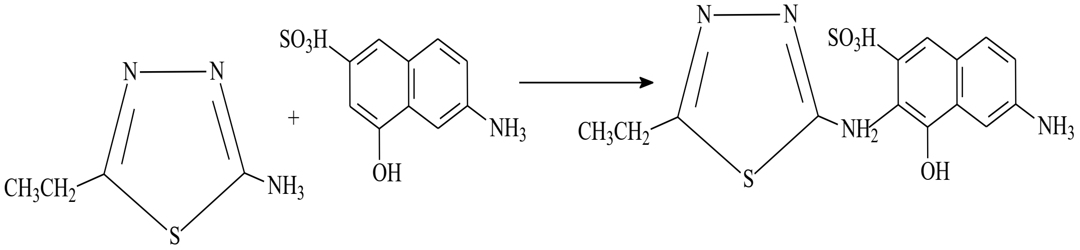
Scheme 1: Diazonium salt of 2-aminothiozole.



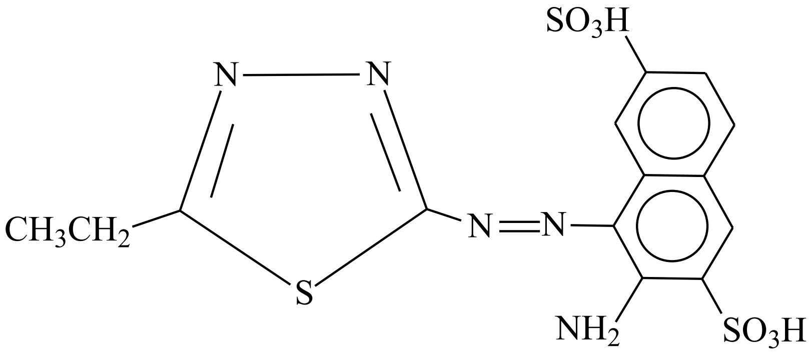
Scheme 2: Diazonium salt of 2-aminothiadiazole.



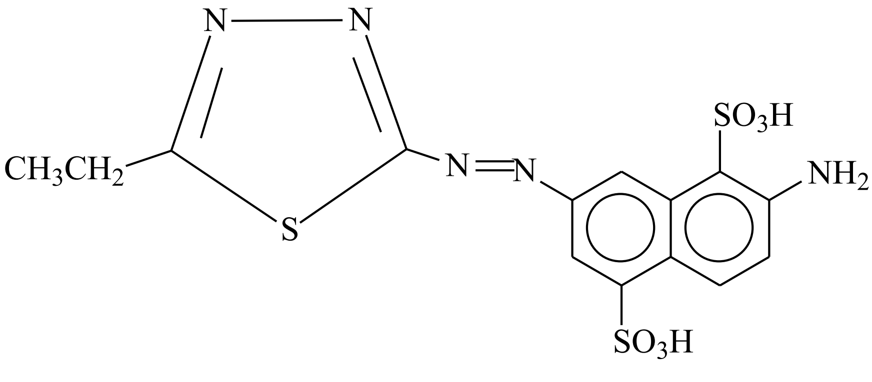
Scheme 3: Coupling Reaction of 2-aminothiadiazole with H-acid. Dye 1



Scheme 4: Coupling Reaction of 2-aminothiadiazole with Gamma acid Dye 2



Scheme 5: Coupling reaction of 2-aminothiozole with R acid Dye 3



Scheme 6: Coupling reaction of 2-aminothiadiazole with Tobias acid Dye 4

Diazotization

5-Ethyl-1,3,4-thiadiazol-2-amine (0.002 1, 2 g molar) was dissolved in hot glacial acetic acid and propionic acid, and the mixture (2:1.6.0 m) was rapidly cooled in an ice/salt bath to 5 °C. The liquor was then added to a cold solution of nitrosyl sulphuric acid prepared from sodium nitrite (0.15 g) and concentrated sulphuric acid (3 ml at 50 °C) over 30 minutes. The mixture was stirred for an additional 2 hours at 0 °C, and excess nitrous acid was dissolved by the addition of urea. The resulting diazonium salt was cooled in a salt/ice mixture (Scheme 2).

Coupling

After diazotization was completed, the diazo liquor was slowly added to a vigorously stirred solution of BON acid in potassium hydroxide (2.0x10^3 mol) and water (20 ml). The solution was stirred at 0-5 °C for 2 hours. After 2 hours, the pH of the reaction mixture was maintained at 4-6 by the simultaneous addition of saturated sodium carbonate solution. The mixture was stirred for one day at room temperature. After 1 day, the resulting solid was filtered, washed with cold water, and dried. The same procedure was repeated for H-acid (Scheme 3), Gamma acid (Scheme 4) R acid (Scheme 5) and Tobias acid (Scheme 6).

Characterization of the Dyes.

Melting Point Determination

The melting points of each dye were determined using a Gallenkamp melting apparatus. A small amount of each dye was filled into a capillary tube and placed into the apparatus. The melting points of each sample were determined by continuously monitoring the apparatus as it gradually heated the samples to assess the purity of the dyes (Nasir *et al.*, 2021).

UV-Visible Spectroscopy

Visible absorption spectra of the dyes were recorded on an Agilent Cary 300 UV-Visible Spectrophotometer from dye solutions in ethanol at a given concentration.

FT-IR Spectroscopy

The infrared spectra of the intermediate and the synthesized dyes were recorded using an Agilent Cary 630 FT-IR spectrophotometer to identify the functional groups present in both (Nasir *et al.*, 2021).

Dyeing of the Fabric (Nylon 6 6)

1% stock solution of each dye was prepared; an aliquot ratio of 50:1 was used, with 2% shade on weight of fabric (o.w.) and 1 g of fabric (Nylon 6 6). The volume required from each stock solution was calculated based on the formula below:

V-WxP/C

Where V is volume required from stock

W is weight of the nylon 6 6

P is percentage shade

V is concentration of the dye (1 g)

Dyeing was carried out on chrome-tanned leather using all the dyes synthesized in the laboratory at a liquor ratio of 1:50. A stock solution (2%) of each dye was prepared, and 5 cm^3 was taken from each dye solution and added to the dye bath containing a small amount of dispersing agent (2 cm^3). Water was then added to make the dye bath up to 50 cm^3. After the dye bath was set up, the fabrics (2 g each) were wetted, excess water was squeezed out with filter paper, and the fabrics were introduced into the dye bath at 50 °C. The temperature was raised to boiling over 15 minutes, and dyeing continued at this temperature for 1 hour (Jitender *et al.*, 2020).

Determination of Light Fastness

An artificial light source (xenon arc) was used to determine the light fastness values of the dyed leather. Strips of the dyed materials were arranged on a card alongside strips of blue wool standards for light fastness testing (numbers 1–8). Seven cards were made for three dyed materials. Each card was placed within a glass frame and screwed into a rigid steel frame. One side of the material was covered with opaque cardboard to shield it from exposure to light. Each glass casing had a small reservoir screened into its base to attain optimum conditions. Exposure of the dyed materials to the light source continued until the most resistant material on each card faded to an extent equal to the contrast rating of 5 on the grey scale. Light-fastness values for each material were determined by comparing the degree of fading with that observed in the blue wool standard, and ratings were assigned accordingly (Silverstein, 2015).

Determination of Wash Fastness

All dyed materials were assessed for wash fastness according to ISO No. 3. The soap solution contained 5 g of Lux soap per liter and 2 g of sodium carbonate per liter of distilled water. The dyed fabric was cut (2 cm × 5 cm) and placed between two adjacent white fabrics of similar dimensions, stapled at all four edges to form a composite specimen. The composite specimen was treated at 60 °C for 30 minutes. After washing, the fabric was removed, separated from the white fabric, dried, and analyzed for colour change and staining of the adjacent white material using a grey scale (Nasir *et al.*, 2021).

RESULTS AND DISCUSSION

The molecular weight, melting point, percentage yield, and colour of the synthesized dyes and the intermediate are presented in Table 1.

Table 1: Physical Properties of the Synthesised Dyes

Dye No.	Melting Point (°C)	Dye Color	Yield (%)
D1	167–169	Orange	82.2
D2	177–179	Purple	86.6
D3	187–177	Pink	87.3
D4	164–165	Pale Brown	90.2

The physical characteristics of the dyes are shown in Table 1. The dyes are in crystal form, with colours ranging from black to orange-yellow. The dyes on the fabrics are dark and orange. The percentage yields of the dyes were also good, with the highest being dye D2 (86.6%) and the lowest being dye D1 (82.2%). The dyes' melting ranges are D1: 167-169 ºC and D2: 177-179 ºC, respectively. The factors that can affect the max value of a dye are the presence of electron-donating or electron-withdrawing groups in the dye molecule, which can shift the max value to longer or shorter wavelengths, respectively.

Visible Absorption Spectra of Dyes

The visible absorption spectra of the dyes were measured in ethanol, DMF and distilled water. The results are presented in Table 2.

Table 2: UV-Visible Properties of the Dyes

Dye No.	Methanol λmax (nm)	DMSO λmax (nm)
D1	447	614
D2	482	483
D3	482	581
D4	530	672

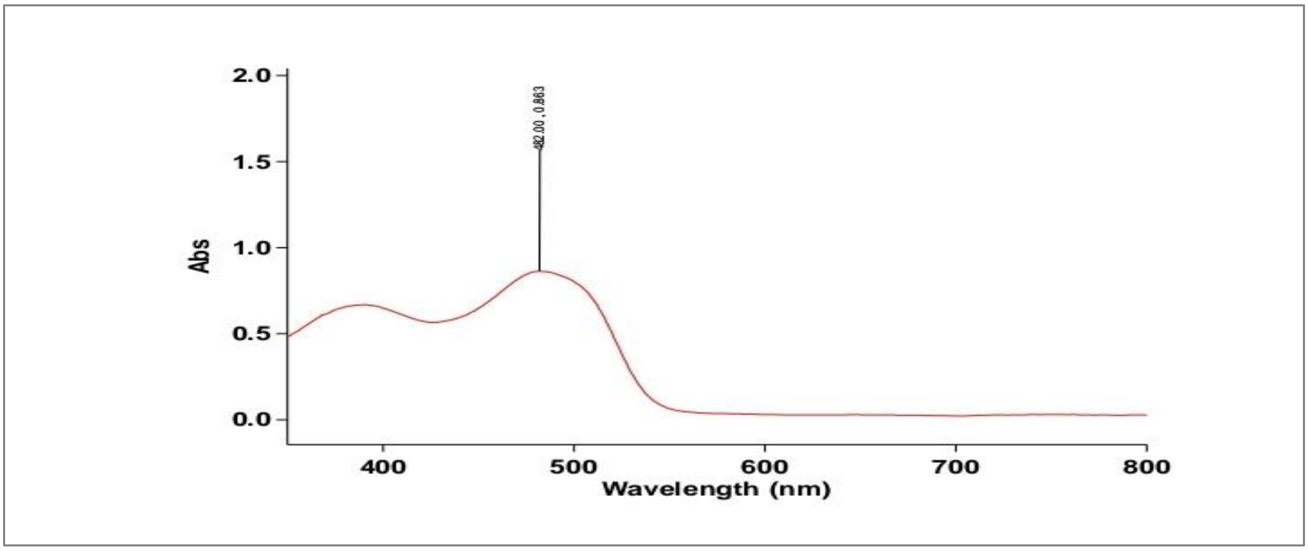


Figure 1: UV-Visible spectra of synthesized Dye 1

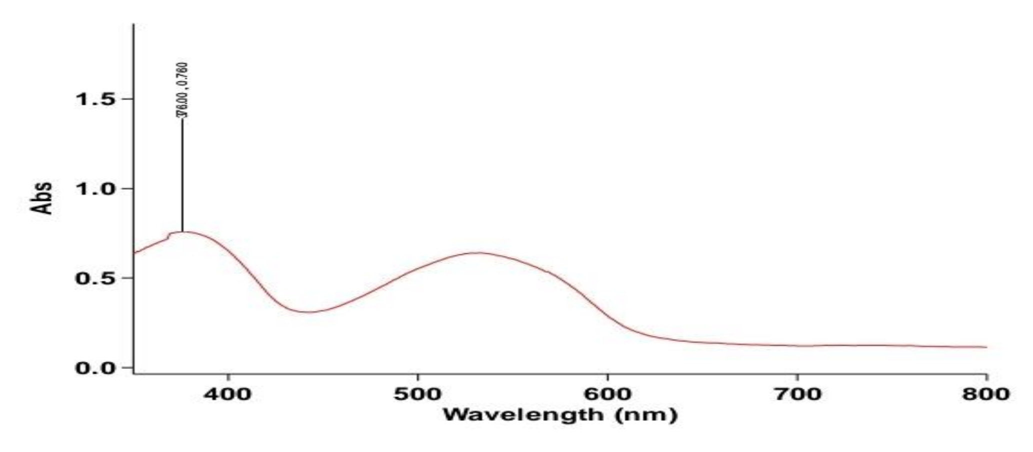


Figure 2: UV-Visible spectra of synthesized Dye 2

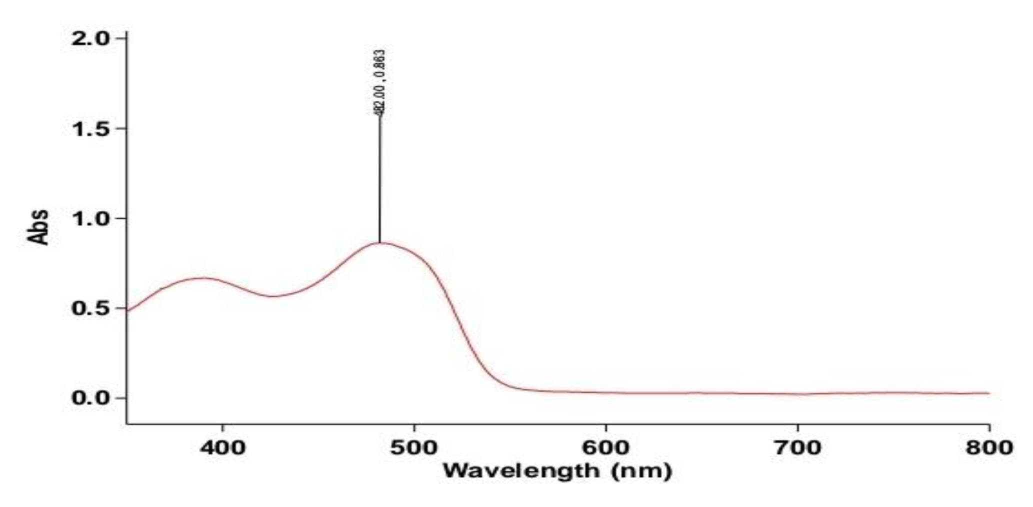


Figure 3: UV-Visible spectra of synthesized Dye 3

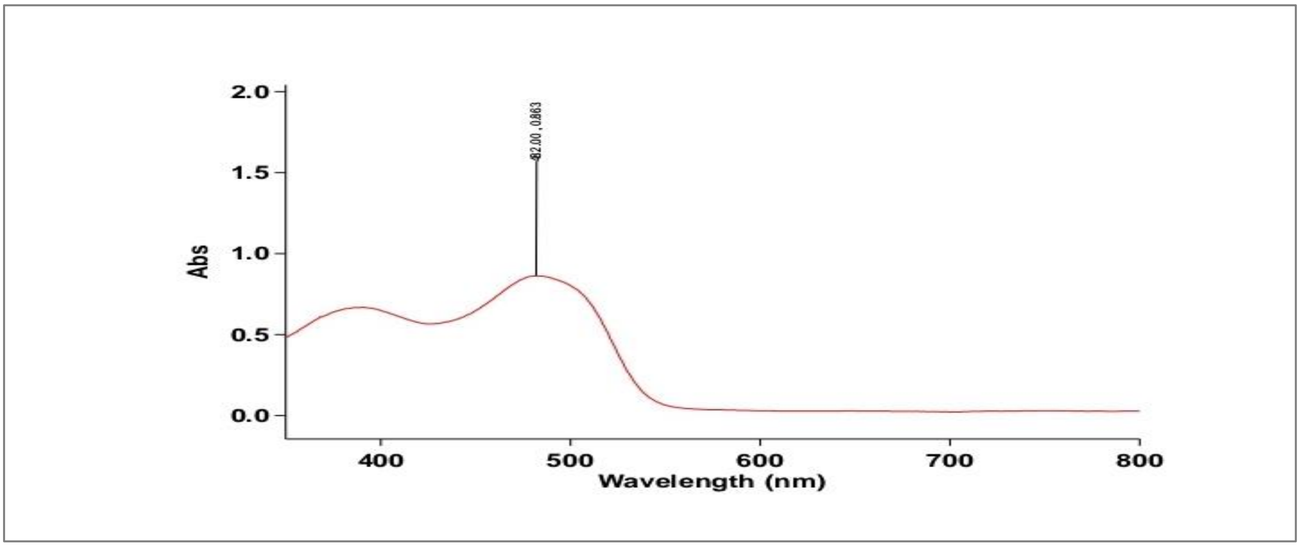


Figure 4: UV-Visible spectra of synthesized Dye 4

Table 3: FTIR Properties of the Synthesised Dyes

Dye No.	FTIR Absorption Bands (cm⁻¹)
D1	3365.8 (O–H / N–H stretch), 1632.8 (C=N), 1513.3 (N=N), 1278.5 (C–S)
D2	3362.1 (O–H stretch), 1632.6 (C=N), 1513.3 (N=N), 1278.5 (C–S)
D3	3369.5 (N–N stretch), 2617 (O–H stretch), 1617.7 (C=C), 1576.7 (N=N)
D4	3362.1 (O–H stretch), 26017 (O–H stretch), 1513.3 (N–NH stretch)

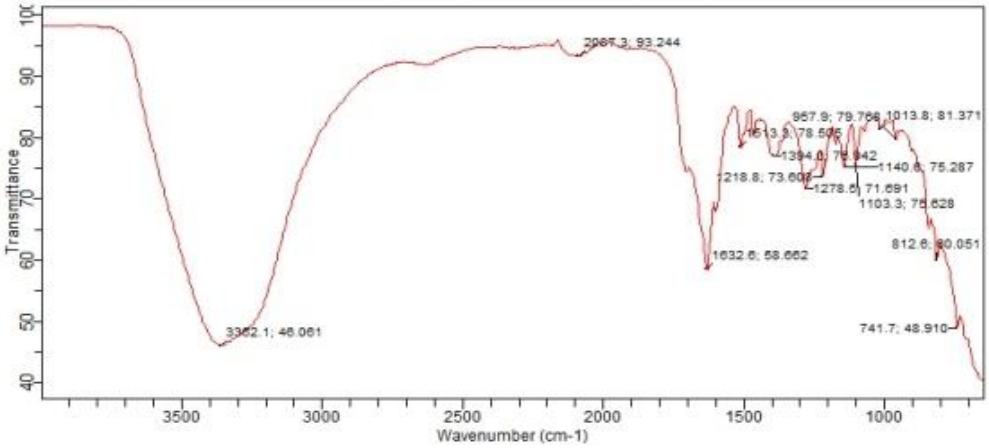


Figure 5: FTIR spectrum of synthesized Dye 1

The UV-Visible spectral data (Figure 1, 2, 3, and 4) presented in Table 2 show the maximum absorbance wavelengths (λmax) of various dyes (D1, D2, D3, and D4) in two solvents: methanol and DMSO (dimethyl sulfoxide). The λmax values indicate the wavelengths at which each dye exhibits maximum light absorption, which is crucial for understanding their electronic transitions and potential applications in fields such as dye-sensitized solar cells, photodynamic therapy, and colourants. The λmax values for the dyes differ significantly between methanol and DMSO. For instance, D1 shows a λmax of 447 nm in methanol and 614 nm in DMSO. This substantial shift suggests that the solvent environment has a pronounced effect on the dye molecules' electronic structure. DMSO, being a polar aprotic solvent, may stabilize certain electronic states differently than methanol, leading to red shifts (longer wavelengths) in the absorption spectrum. The observed shifts can be attributed to solvent-solute interactions, such as hydrogen bonding and dipole-dipole interactions, which can influence the energy levels of the electronic transitions. Dyes D2, D3, and D4 exhibit similar λmax values in methanol (482 nm for D2 and D3) and a notable increase in λmax in DMSO (483 nm for D2 and 581 nm for D3, and 672 nm for D4). The consistent λmax for D2 and D3 in methanol suggests that they may have similar electronic structures or chromophores. D4, with a λmax of 530 nm in methanol and a significant increase to 672 nm in DMSO, indicates a strong solvent effect and possibly a more complex electronic structure that is more sensitive to the solvent environment.

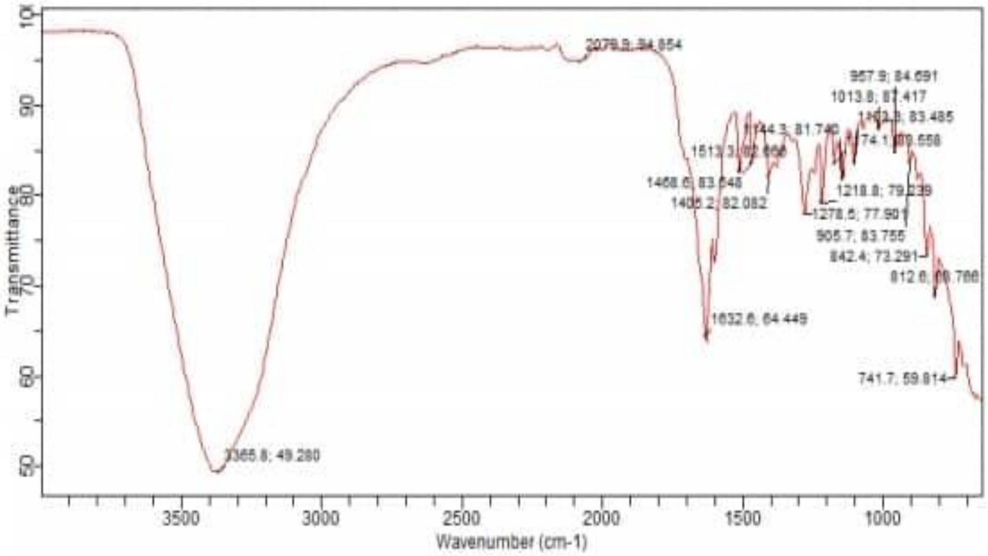


Figure 6: FT-IR spectra of synthesized Dye 2

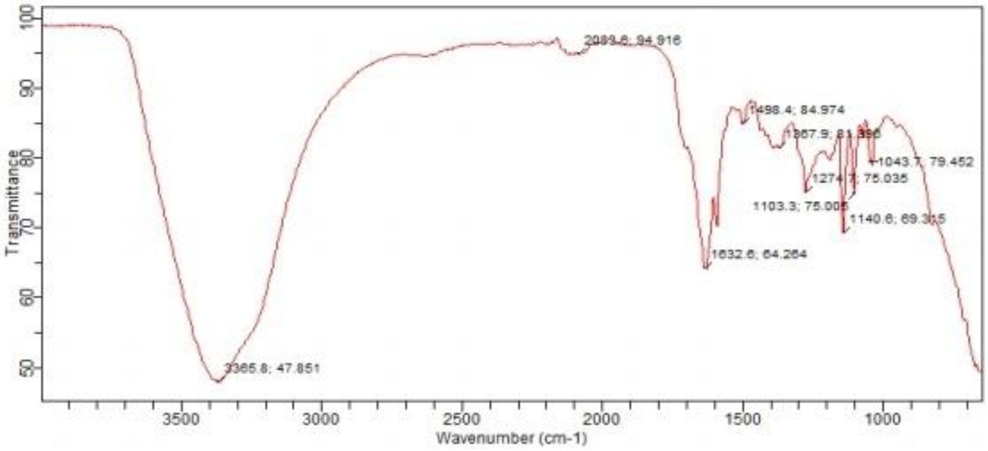


Figure 7: FT-IR spectra of synthesized Dye 3

Infra-Red Spectra of the Synthesized Dyes and Metalised Dyes

IR spectroscopy is often used to identify structures because functional groups give rise to characteristic bands in terms of both intensity and position (frequency), as shown in Table 3.

The vibrational spectrum of a molecule is considered to be a unique physical property and is characteristic of the molecule. As such, the infrared spectrum can be used as a fingerprint for identification by comparing the spectrum of an "unknown" with previously recorded reference spectra. The infrared spectrum is formed as a consequence of the absorption of electromagnetic radiation at frequencies that correlate to the vibration of specific set of chemical bonds from within a molecule. The FT-IR functional group vibrations of spectra of synthesized dyes (Figure 5, 6, 7, and 8) are shown in Table 3. The FT-IR spectrum shows the different functional groups present in each dye molecule. D1 and D4 both have 3365.8(OH/NH) groups, as well as 1632.8 (C=N) and1632.6 (C-S) bonds. D1 has an additional 1513.3 (N=NH) bond, while D4 has an extra OH group. D2 has 3362.1 (OH), 1513.3(N=N), 1278.5 (C=N), and 1278.5

(C-S) bonds, while D3 has 3369.5 (N-N), 3362.1 (OH), 1576.7(N=N), and 1617.7 (C=C) bonds. The synthesized dyes possess various functional groups that can influence their spectroscopic properties and potential applications.

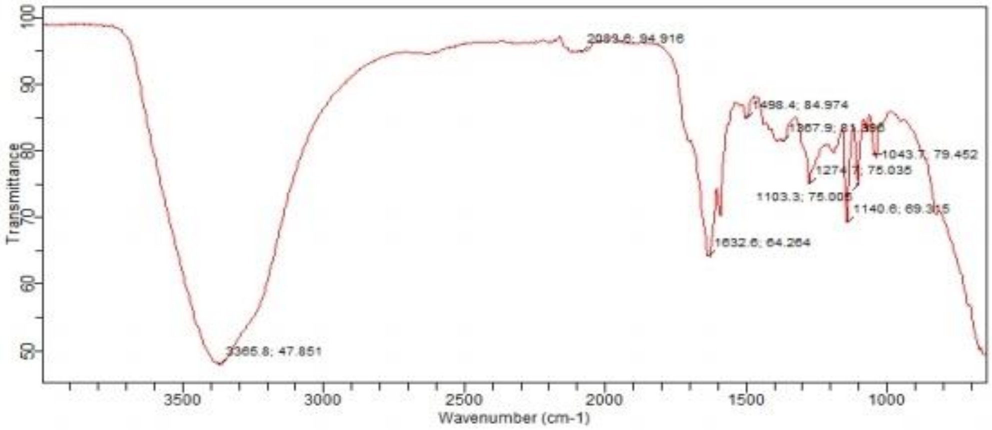


Figure 8: FT-IR spectra of synthesized Dye 4

Table 4: Wash and Light Fastness of the Dyed Leather

Dye No.	Change in Shade	Staining	Light Fastness	Color on Fabric
D1	5	5	7	Orange
D2	4/5	4/5	6	Purple
D3	4/5	4	5	Pink
D4	2	3/4	5	Pale Brown

Wash fastness (Change in Shade & Staining): 5 = Excellent, 4/5 = Very Good to Excellent, 4 = Very Good, 3 = Good, 2 = Fair, 1 = Poor (Grey Scale).
Light fastness: 8 = Outstanding, 7 = Excellent, 6 = Very Good, 5 = Good, 4 = Moderate, 3 = Fair, 2 = Poor, 1 = Very Poor (Blue Wool Scale).

The presence of specific bonds, such as N=N and C=C, could indicate potential for colour stability or other desirable properties in dyeing applications. The differences in the FT-IR spectra of the dyes suggest they have unique chemical compositions, which could lead to differences in their performance as dyes. Further analysis and testing would be needed to characterize and understand the spectroscopic properties of these dyes fully.

Wash and Light Fastness of the Dyed Leather

The wash and light fastness of the dyed leather were evaluated using a standard procedure, and the results are presented in Table 4.

The results of the wash and light fastness tests on the dyed leather show that D1 has excellent wash and light fastness, with a change in shade rating of 5, staining rating of 5, and light fastness rating of 7. The color on fabric remains orange. D2, D3, and D4 all show lower ratings in terms of wash and light fastness. D2 and D3 have a change in shade rating of 4/5, a staining rating of 4/5, and a light fastness rating of 4/5. However, D4 has a lower change in shade rating of 2, a staining rating of 4, and a light fastness rating of 3/4. The colors on fabric for D2, D3, and D4 are purple, pale brown, and pink, respectively. It can be concluded that D1 performs the best in terms of wash and light fastness, while D2, D3, and D4 show slightly lower ratings in these areas. Further improvements may be needed in the dyeing process for D2, D3, and D4 to enhance their wash and light fastness properties.

Light fastness rating of the dyed samples is shown in Table 4. The test was carried out by exposing each dyed sample to daylight alongside eight blue wool standards. It was observed that the light fastness of nylon 6,6 was higher due to the nature of the fabric used. This may be attributed to the molecular structure of the dye and its substituents, which provide shielding from radiant energy for the azo chromophore (Aljamali, 2017). Consequently, this effect conferred good light fastness properties on the dye (Obadahun *et al.*, 2020). The results show that the fastness level of dye sample D4 indicated very slight alteration in shade or a slight loss in depth of colour. The fastness rating was 4, corresponding to slight colour loss. This may be due to the lower surface area-to-volume ratio, implying that less dye was present on the surface. The rate of fading was therefore higher for filaments and microfibers (Obadahun *et al.*, 2023).

CONCLUSION

In conclusion, the substituent effect on the absorption spectra of 8-amino-2-hydroxynaphthalene-1,6-disulfonic acid-1,3,4-thiadiazole has been shown to affect the compound's absorption properties significantly. Different substituents at different positions can shift the absorption maxima and alter the intensities of absorption bands, highlighting the importance of understanding the molecule's electronic structure for tailoring its optical properties.

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