DOI: 10.67867/ccj.2026v10i3.12

September 2026; 10(3): 111-119

ChemClass Journal

Publication of the Chemical Society of Nigeria, Zaria Chapter

Journal homepage: https://chemclassjournal.com.ng

e-ISSN: 3092-8214; p-ISSN: 3092-8206

Research Article

Synthesis and application of novel thiophene-based monoazo acid dyes on chrome-tanned leather and nylon 6,6 fabrics

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Article History
Received: June 3, 2026
Reviewed: June 12, 2026
Revised: July 5, 2026
Accepted: August 17, 2026
Published: September 30, 2026

Citation
Adeyi A. O., Habila B., Adeoye D. O., Abolude I.O., Magaji I.Y. (2026). Synthesis and application of novel thiophene-based monoazo acid dyes on chrome-tanned leather and nylon 6,6 fabrics. *ChemClass Journal,* 10(3), 111–119. DOI: 10.67867/ccj.2026v10i3.12

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ABSTRACT

Four novel mono azo acid dyes have been formed in moderate yield from diazotization of 2-aminothiophene -4-carboxylic acid and coupling with cyanurated 2-naphthol-3-6 disulforic acid (R acid), 8–amino–naphthol 3-6-disulfuric acid (H acid), 2-amino -5- naphthol -7- sulfuric acid (J acid) and 2-amino -1- naphthalene sulfuric acid (Y acid). The synthesized dyes gave a percentage yield ranging from 62-82, melting point 120-214°Cand molecular weight 507-587 g/mol. The dyes were characterized by analytical technique such as FTIR, UV-visible and GCMS of intermediate. The dyes were found to give shade of red dark blue and darkmaroon .The synthesized dyes gave a maximum absorption within the UV- visible region with λ_{max}530-597 nm which indicate bathochromic effect and molar extinction coefficient ranging from (3.217-4.221 x 104 l/mol^{-1} cm^{-1}) and the infra-red spectra showed peaks at 1582-1421cm-^{1} (due to azo group (N=N) stretching vibration), at 3400 – 3200 cm-1 due to (OH&NH) medium & broad stretching vibration), at 2830-26-95cm-1 (due to aromatic group (C-H) stretching vibration) , at 1760-1640 cm-1 (due to carbonyl group (C=O) stretching vibration), at 1342-1266 cm^{-1} (due to aromatic amine group (C-N) stretching vibration) and at 2260-2222 cm-^{1} (due to nitrile (C≡N) stretching vibration). The dye exhibited good depth, affinity, levelling and brightness properties on the substrates. The dye gave a good percentage exhaustion on nylon 6.6 and on chrome tanned leather substrates ranging from (78-87%) and (63-75%) respectively was successfully synthesized. The chrome tanned leather gave a wash fastness rating ranging from (3 -5) good, very good to excellent respectively and nylon 6,6 gave a wash fastness rating ranging from (4-5) very good to excellent fastness properties and the light fastness properties of chrome tanned leather gave a fastness rating (4-7) moderate, good, very good and excellent and rubbing fastness rating (4-5) very good to excellent on both substrates. The good shades observed may be due to better substantivity on chrome tanned leather and nylon 6, 6 fabrics. These findings indicate that the dyes possess high affinity for the chrome tanned leather and nylon 6, 6, good stability of shade development, and high stability under textile processing conditions. The study was confined to nylon 6.6 under laboratory conditions, and further work is needed to test its behaviour on other polyamides and to assess its environmental performance.

Keywords: Synthesis, Diazotization, Analytical technique, Colour strength, Fastness properties

Introduction

Synthetic organic colorants form the basis of the aesthetic and functional performance of thousands of products in many industrial sectors, such as textiles, paper, plastics, leather goods, coatings, cosmetics, and food. Ever since the introduction of the first synthetic dyes in the mid-nineteenth century, their synthetic molecules have largely replaced natural colorants due to their superior performance attributes, palette of colors offered, improvements in fastness properties, and economies of scale realized through chemical synthesis. It is also possible to customize the molecular structure, resulting in the production of dyes with targeted affinities to other substrates and performance profiles [1, 2].

Amid the vast array of synthetic dyes, the azo group is the most commercially important and most commonly used class of organic dyes. Azo dyes (termed as such by the presence of one or more azo chromophores (-N=N-) connecting aromatic or heteroaromatic groups) make up a significant proportion of the total world dye market, encompassing a significant majority of all the organic dyes produced and used worldwide, estimated at 60-70%. These dyes are usually easy and cheap to synthesize, and the most common method is the diazotization of a primary aromatic amine in an azo coupling reaction, with an electron-rich coupling partner, which can be a phenol, naphthol, or an amine [3]. This synthetic flexibility permits the production of a vast structural variety which may be tailored to yield almost any color, including the entire visible spectrum. Moreover, numerous azo dyes have high values of molar extinction coefficient, which is equivalent to high tinctorial power, and moderate to good fastness to light, washing, and perspiration, which implies that azo dyes can be used in a wide variety of demanding applications [6]. Azo dyes are grouped by the number of azo groups (monoazo, disazo, trisazo, polyazo) or by application method/substrate affinity, resulting in such groups as acid, basic, direct, disperse, and reactive dyes. This success and synthetic accessibility, which constitute the basis of the dominance of azo dyes, testify to a technologically mature discipline in which common synthetic pathways are highly optimized towards large-scale manufacture [7]. The use of unusual, structurally resilient structural motifs or functional groups provides a potential direction of the synthesis of the next generation of dyes with specific characteristics. At the same time, mass production and use of synthetic dyes, such as the azo group, present environmental concerns regarding manufacturing and effluent discharge. The potential negative impacts of specific dyes and their by-products on human health, as well as the intensive use of water in traditional dyeing techniques, have led to the development of more environmentally friendly alternatives [12]. This involves developing greener dyeing processes (such as supercritical carbon dioxide or natural colorants) and creating dyes with enhanced fixation capabilities, thereby reducing the quantity of residual colorants discharged into wastewater [5]. As such, the improvement of dye performance, especially its fixation and fastness, is both technically and environmentally desirable. The aim of the study was to synthesis novel monoazo acid dyes and the assessment of it fastness properties on chrome tanned leather and nylon 6,6 derive from 2-aminothiophene-4-carboxylic acid using coupling component such as 2-naphthol-3-6 disulforic acid (R acid), 8–amino–naphthol 3-6-disulfuric acid (H acid) , 2-amino -5- naphthol -7- sulfuric acid (J acid) and 2-amino -1- naphthalene sulfuric acid (Y acid). Which was achieved through the following objectives: Synthesized of aminothiophene , To synthesize monoazo acid dyes derived from 2-aminothiophene,To study the spectroscopic properties of synthesize dyes and to utilised the synthesized dye in the coloration of chrome tanned leather and nylon 6,6 substrates.

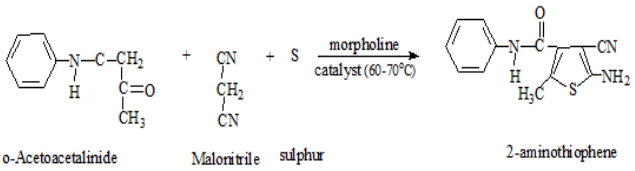
Material and Methods

Materials

All commercial products were purchased from Sigma-Aldrich. Such as cyanuric chloride, vinyl sulphone, 2-naphthol-3-6 disulforic acid (R acid), 8–amino–naphthol 3-6-disulfuric acid (H acid) , 2-amino -5- naphthol -7- sulfuric acid (J acid) and 2-amino -1- naphthalene sulfuric acid (Y acid) , acetic acid ,anionic detergent FT-IR spectrophotometer (Agilent CARRY 630 FT-IR Spectrophotometer) Ahmadu Bello University , GC-MS spectrometry (7890B GC System) Ahmadu Bello University, tanning vessels NILEST Zaria, rub fastness tester NILEST Zaria, retort stand, sample bottle, water bath, ice bath cornical flask.

Synthesis of dye intermediate (2-Aminothiophene intermediate)

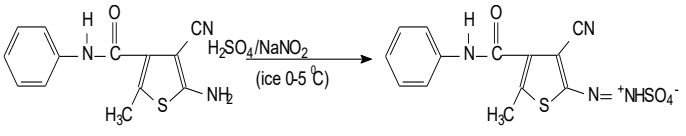
The dye intermediates was synthesized as follows: o-acetoacetanilide (20.0 g, 0.1 mol), Malononitrile (6.50 g, 0.1 mol), Sulphur (3.0 g, 0.1 mol), morpholine (8.50 g, 0.1 mol) were combined in the presence of 200 cm3 of ethanol were introduced into a round bottom flask and place on a heating mantle at 60-70 o C on stirring condition for 3-4 hrs. The resulting thick dark solution is cooled and store overnight in a refrigerator, followed by purification, washing with ethanol and then 50 cm3 ethanol and 10 cm3 of distilled water solution and wash dried at room temperature. The light brown powder obtained was crystallized from ethanol [8]



Scheme 1. Schematic route for the synthesis of intermediate (Aminothiophene)

Synthesis of diazonium salt solution [8]

Sodium nitrite (3.0 g, 0.02mol) is added portion to 50cm^3 of concentrated sulphuric acid at 10^{o}C and heated to 60 ^{o}C with stirring for 15 min. The solution was then cool to 0-5 ^{o}C in ice bath and a mixture of 20cm^3 acetic acid and 10 cm^3 propionic acid (2:1) is then added to the 18g synthesized intermediate. The reaction mixture was then cooled to 0-5 ^{o}C and is then added portion wise and stirring is continued for 2 hrs at 0-5 ^{o}C. The resulting diazonium salt solution is obtained.

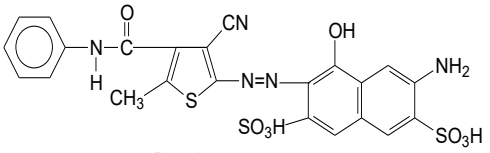


Scheme 2. Schematic route for the synthesis of diazonium salt.

Coupling of diazonium salt solution [4]

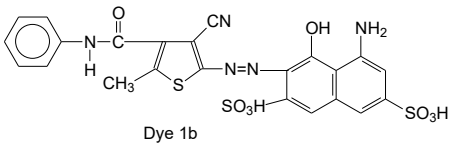
H-acid (2.0g, 0.01 mole) is dissolved in 10 cm^3 of distilled water and the solution was cool to 0-5^{o}C. To the well stirred solution,

the freshly prepared diazonium salt solution was added drop wise over 45 minutes maintaining the temperature below 0-5 °C with vigorous stirring. The stirring was done for 1 hour at 0-5 °C, maintaining pH 4-5 using sodium carbonate solution (10%$^{w}/_{v}$). The resulting dye was collected, wash several times with water to ensure a free acid and dry in an oven at 40 °C. The result was repeated for 2-naphthol-3-6 disulforic acid (R acid), 8–amino–naphthol 3-6-disulfuric acid (H acid) , 2-amino -5- naphthol -7-sulfuric acid (J acid) and 2-amino -1- naphthalene sulfuric acid (Y acid) .



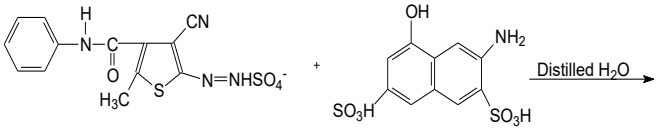
Dye 1a

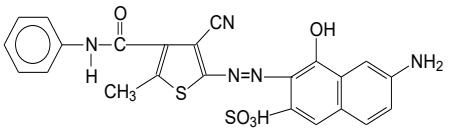
3-amino-6-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)naphthalene-2,7-disulfonic acid



Dye 1b

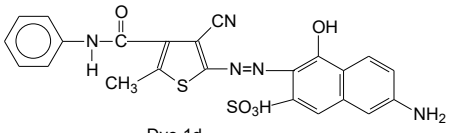
5-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2,7-disulfonic acid





Dye 1c

6-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2-sulfonic acid



Dye 1d

7-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2-sulfonic acid

Structures of Synthesized Dyes

The various schemes above show the synthesis of dye 1a, 1b, 1c and 1d respectively with the coupling component

Purification of the Dyes

The dyes were purified through the same process of recrystallization as highlighted above for the intermediates. Some of the dyes required the mixed solvent of ethanol, methanol /DMF (9/1 solvent mixture) according to a procedure suggested by [9]. The purity of each synthesized dyes were confirmed by melting point.

Percentage Yield of Dyes and Intermediates

The percentage yield of the synthesized dyes and intermediates were determined using the formula shown below [9]

$$\%\text{Yield} = \frac{MP}{MMP} \div \frac{MR}{MMR} \times 100$$

where;
MP is the mass of the product
MMP is the molar mass of the product
MR is the mass of the reactant
MMR is the molar mass of the reactant

Melting point of the dyes

The melting point of each dye was determined using Gallenkamp melting apparatus. Small amount of each dye was filled into a capillary tube each placed in to the apparatus, the melting point of each dye was obtained by consistently focusing on the apparatus as the apparatus gradually heat the dye in the tube where the melting point is obtained. [4]

Dyeing of nylon 6, 6 fabric

For the nylon 6, 6 fibre, a dispersing agent (anionic detergent) was used to facilitate the dyeing process. The fabric was wetted and thoroughly squeezed to remove excess water. It was then immersed into the bath at 70°C and allowed to reach the boil within 15 min. Dyeing was carried out for one hour at a temperature of 100°C with agitation. At the end of the dyeing process, the substrate was removed, squeezed and rinsed thoroughly under running tap water and allowed to dry at room temperature.

Dyeing of leather

The synthesized dyes were used in dyeing of chrome tanned leather. The standard method of dyeing leather was followed using the recipe.

i. 120 % of water (60 ^{0}C)
ii. 2 % of dye
iii. % of formic acid

A solution of the dye sample (2%) was made with distilled water using heating mantle and the temperature of the medium was raised to 60 ^{0}C. The pH of the bath was adjusted to 5.5 with formic acid. The leather samples were introduced into the bath and run for 1 hour in a mechanical shaker at a controlled speed [9]

Application of dyes solution

A solution was prepared by dissolving about 1g of dried dye powder in 100mls of distilled water. To determine the qualities required for the experiment the following parameter was utilized.

$$\text{Number of ml of stock solution required} = \frac{W \times P}{C}$$

Where W = weight (in grams) of sample to be dyed
P = percentage of dye to be used
C = concentration (%) of stock solution [11]

General procedure of dyeing chrome tanned leather

1 % of dye (on weight of the materials)
50 + 2°C dyeing temperature
0.1 % of formic acid on weight of the dye
Time of dyeing: one hour
Liquor ratio 20:1

Determination of dye bath exhaustion

Dye uptake was determined by measuring the absorbance of diluted dye bath samples at the wavelength of the maximum absorption. The bath was sampled before and after dyeing. A 1ml aliquot was taken from the bath and diluted in 20ml of distilled water. This is to ensure that the absorbance falls within the readable range of 1.5. The percentage dye bath exhaustion (%E) for each substrate was calculated using the equation below [11].

$$\% E = \frac{Ao - A1}{Ao} \times 100$$

Where Ao and A1 are the absorbance at λmax of the dye bath prior to dyeing and after dyeing respectively.

Light fastness of dyed substrates (Fastness to Sunlight) SLT 401 (IUF 401)

This test was carried out in accordance with the Society of Leather Technology and Chemists. The test is carried out on the dyed samples under daylight facing south sloping at an angle 45° from the horizontal. The procedures involved aligning both the eight dyed wool standards together with a. 2 cm x 2cm cut pieces of the dyed samples on a card and covering one third of the setup with opaque cover.

Exposure is carried with regular inspection noting when the non-exposed part and exposed part of each dyed sample had contrast equivalent to grade 4 of ISO grey scale after which and opaque cover was placed covering part of the exposed sample part, exposure was continued till the blue standard 7 faded. The rating of the dyed samples is taken to be those equivalents to-that of standard dyed blue wool samples, which faded to the same extent as the specimen at the same time

Wash fastness of the dyed substrates

This is to assess the leather resistance to prolong action of water. 2 x 2cm of the dyed leather was dipped into solution made by 5 g (0.5 %) of detergent powder into 200 cm^3 of distilled water. This was placed in water bath and the temperature allowed to rise to 40 °C. This was vigorously stirred for about 30 min and the leather was transferred to 100 cm3 distilled water for washing and rinsing was done five times and dried. The change in colour was assessed by the standard grey scale.

Rubbing fastness test of the dyed substrates

This method is intended for determining the behaviour of the surface texture of a leather rubbing with felt. The leather to be tested is rubbed with pieces of structure wool felt under a given pressure with a given number of forward and backward motion. 2x2cm piece of the leather sample were cut and dipped on rub fastness tester (Machine Model STM). The number of rub on a fixed position was varied to assess the degree to fastness to rubbing. The change in colour of the felt and leather was assessed with the standard grey scale.

Results and Discussion

Table 2 Display the physical the physical properties of mono azo acid synthesized dyes which comprises the molecular formula , shade of crystal sprang from red, darkblue, dark maroon, molecular weight 507-587 g/mol, melting point ranging from 120-214 °C and percentage yield 62-82% which is according to literature

Table 1. Physical properties of the synthesized dye intermediate

Name of intermediate	Colour	Melting point (°C)	Percentage yield (%)	Molecular weight(g/mol)	Molecular formula
2-Aminothiophene-4-carboxylic acid	Darkbrown	188-194	97	257	$C_{15}H_{11}ON_5S$

Table 2. Physical properties of the synthesized mono acid dyes

Dye no	Molecular formula	Shade crystal	Molecular weight(g/mol)	Melting point (°C)	Percentage yield (%)
1a	$C_{23}H_{17}O_8S_3N_5$	Red	587	200-202	76
1b	$C_{23}H_{17}O_4\ S_5N_5$	Darkblue	587	191-194	62
1c	$C_{26}H_{17}O_5S_2N_5$	Dark maroon	507	120-122	68
1d	$C_{26}H_{17}O_5S_2N_5$	Red	507	212-214	82

Table 3. Spectra data of the synthesized mono acid dyes using dimethylformamide and ethanol.

Dyes no	λ_{max}in (nm) DMF	λ_{max}in (nm) Ethanol	Molar Extinction Coefficient in(DMF) x 10^4 $(l/mol^{-1}cm^{-1})$
1a	530	515	4.181
1b	597	572	4.176
1c	580	577	3.217
1d	565	527	4.145

The spectral data analysis of synthesized monoazo acid dyes

The monoazo acid dye as shown on Table 3: 1a, 1b, 1c and 1d absorbed at 530, 597, 580 and 565 nm respectively in dimethylformamide. Dye 1b for instance is more bathochromic compared with dye 1a, 1c and 1d. dye 1b was synthesized from the coupling component of 8- amino-1- naphthol 3-6-disulfuric acid which contain sulphonic group in it molecules. This is followed by dye 1c containing 2, amino–8–naphtholsulfuric acid as coupling component which absorbed at 580nm and is bathochromic compound with dye 1a and 1d which absorbed at 530 and 565 nm containing 2–naphthol–3–6 disulfuric acid and 2-amino-5-naphthol-7-sulfuric acid respectively, Maximum absorption moves to longer wavelength as the amount of delocalization increases, therefore the maximum absorption

moves to shorter frequency, the absorption needing less energy gap between the bonding and anti-bonding orbitals [8]

Molar Extinction Coefficient (€)

From the result indicated on Table 3: it is clear that the dyes possess high molar extinction coefficient and this may be attributed to the fact that it transmits more light. From the result shown, it can be deduced that majority of the synthesized dyes gave molar extinction coefficient values greater than $4.181\times10^{4}mol^{-1}cm^{-1}$. The higher molar extinction coefficient value of the synthesized dyes probability for more electronic transition and deepness of the shade on the substrate.

Assessment of FT-IR group absorption spectroscopic properties of synthesized intermediate and acid dye

It is obvious as shown on Table 4: that dye intermediate which is 2 – aminothiophere gave absorption peaks due to (N –H) group at 3459.0 cm^{-1}, 3354.6 cm^{-1}, and 3205.5 cm^{-1}, (C – H) group at 2967 cm^{-1}, (C = O) group at 1699.7 cm^{-1}, 1666.1 cm^{-1}, and 1602 cm^{-1}, and (C≡ N) group at 2214.0 cm^{-1}, stretching vibration. Dye 1a – d which is obtained by diazotization of 2–aminothiophene and coupling with 2–naphthol–3–6 disulfuric acid, 8–amino–1–naphthol-3-6, disulfuric acid, 2–amino–8–naphtholsulfuric acid and 2–amino–5–naphthol–7 sulfuric acids respectively. Gave absorption peaks due to (O–H and N–H) group at 3347 cm^{-1}, (C=O) at 1636.3 cm^{-1}, (C–N) at 1315.8 cm^{-1}, and (-N=N-) group at 1488.6 cm^{-1}, stretching vibration in dye 1a. dye 1b which is obtained by diazotization of diazonium salt coupling with 8–amino–1-naphthol–3-6–disulfuric acid showed absorption peaks due to (O–H and N–H) group at 3377.0 cm^{-1}, (C=O) group at 1636 cm^{-1}, (C–N) at 1367cm^{-1}, and (-N=N-) group at 1499.4 and 1529.2cm^{-1}, stretching vibration. Dye 1c which is obtained by coupling diazonium salt with 2–amino–8–naphtholsulfuric acid gave absorption peak due (O – H)\ and N –H) group at 3373.2cm^{-1}, (C=O) group at 1703.4cm^{-1}, and 1654.9cm^{-1}, (C–N) group at 1319.5cm^{-1}, and (N=N) group at 1468.5cm^{-1} stretching vibration. Dye 1d which obtained by coupling diazonium salt with 2–amino–5–naphthol–sulfuric acid showed absorption peak due to (O –H and N – H) group at 3410.5cm^{-1}, (C = O) group at 1636.3 cm^{-1}, (-N=N-) group at 1535.7 cm^{-1}, and 1468.6 cm^{-1}, stretching vibration.

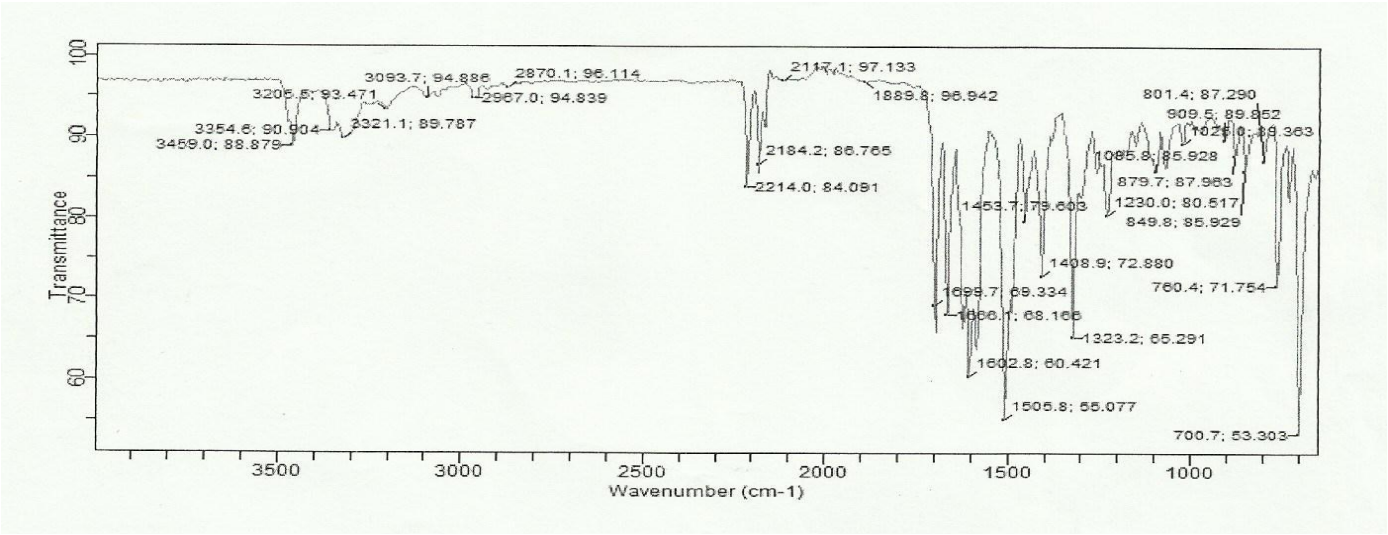
Table 4. Infrared spectra of the synthesized intermediate

Functional group	NH cm^{-1}	C-H cm^{-1}	C=O cm^{-1}	C-N cm^{-1}
Types of vibration	Stretching	Stretching	Stretching	Stretching
	3459.0	2967.0	1699.7	2214.0
	3354.6	2870.1	1666.1	
	3205.5		1602	

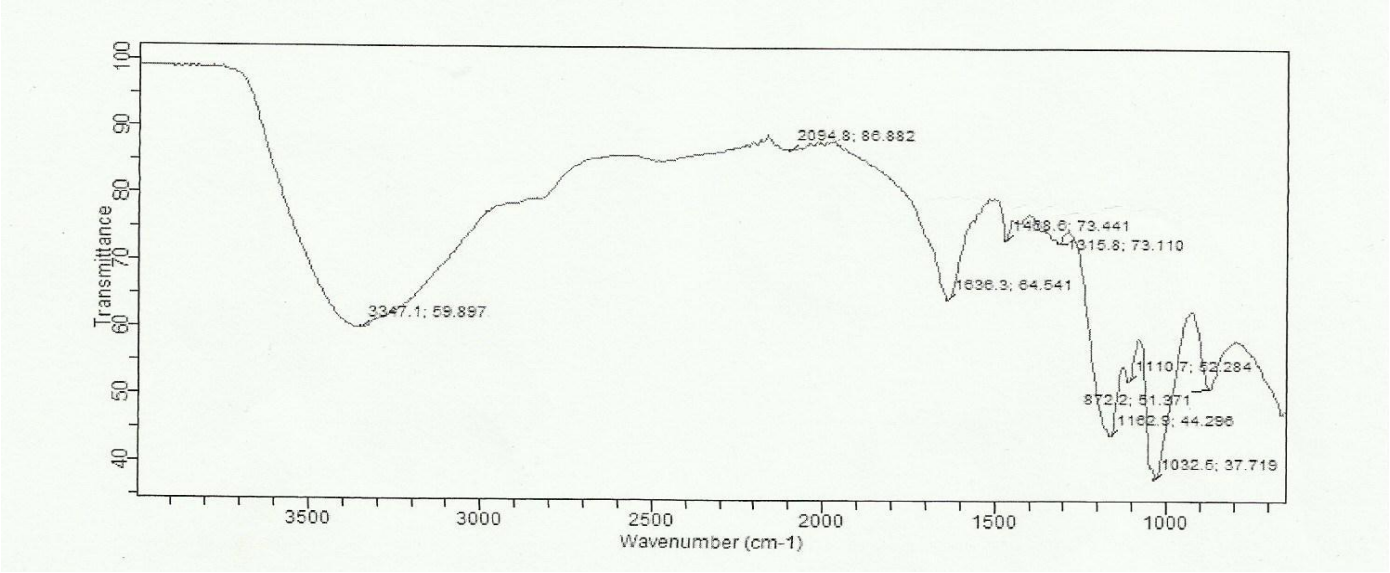
Table 5. Infrared spectra of mono acid dyes

Functional group	OH & NH	SO_3H	Carbonyl C-O	Aromatic amine C-N	Azo N=N
Types of vibration	Stretching	Stretching	Stretching	Stretching	Stretching
1a	3347.1	1150	1636.3	1315.8	1488.6
1b	3377.0	1200	1636.3	1367	1499.4 1529.2
1c	3373.2		1703.4 1654.9	1319.5	1468.5
1d	3410.5		1636.3		1535.7 1468.6

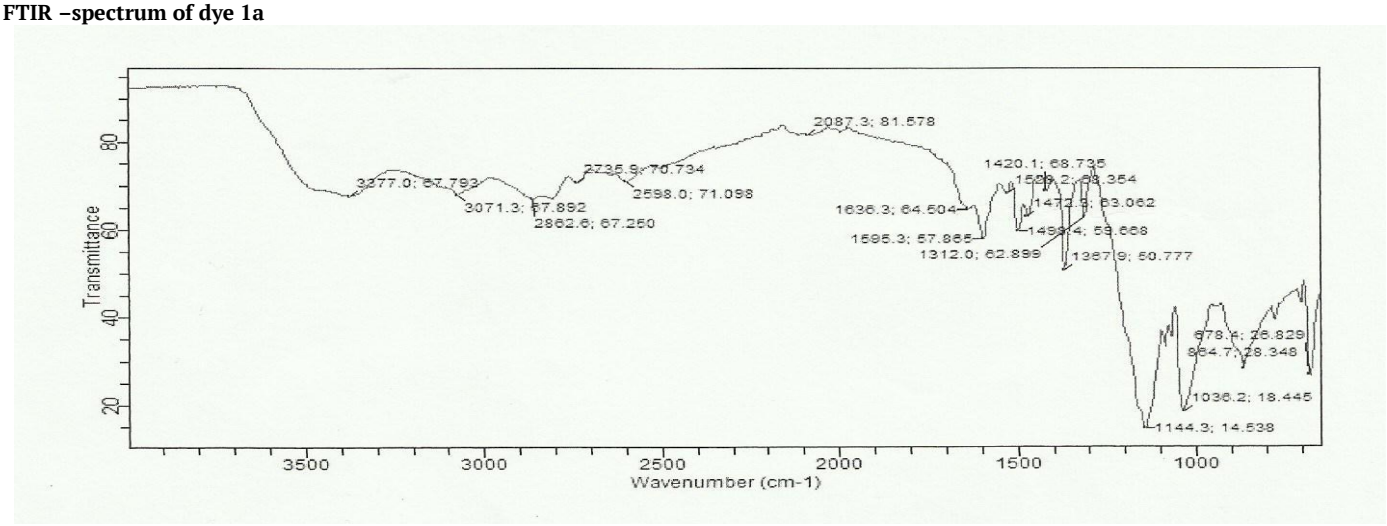
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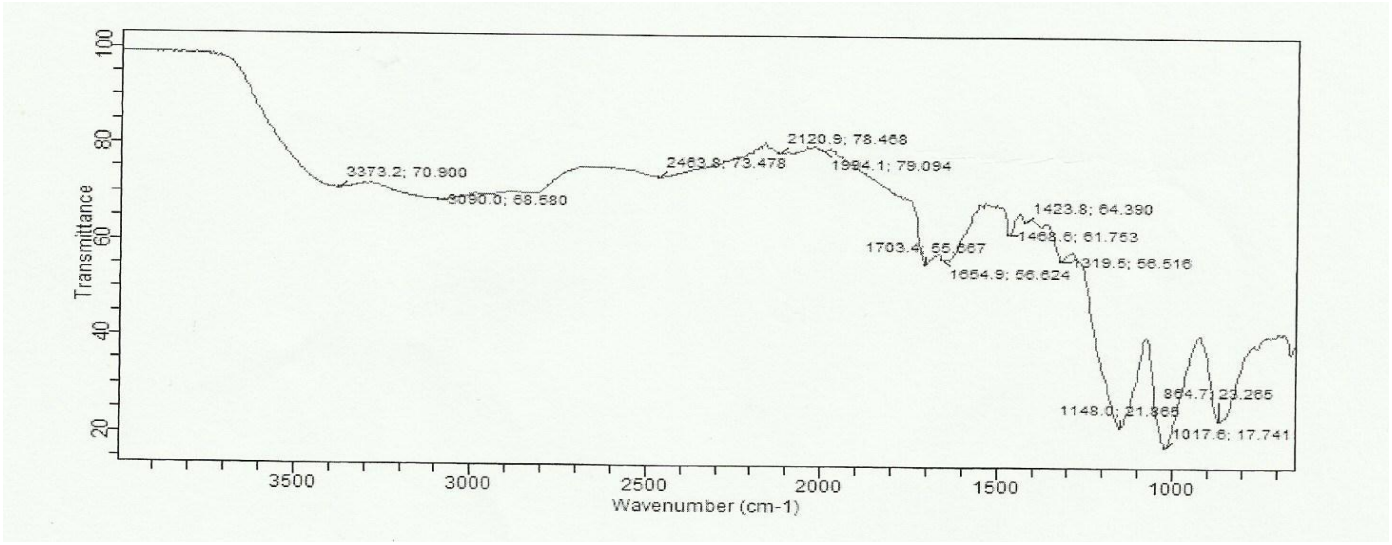
FTIR –spectrum of dye intermediate



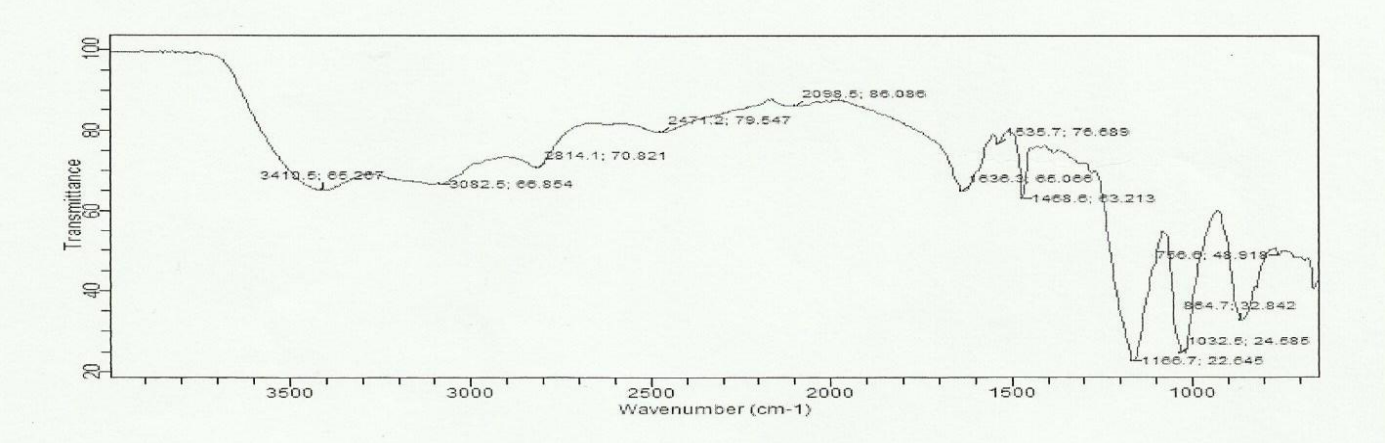
FTIR –spectrum of dye 1a



FTIR –spectrum of dye 1b



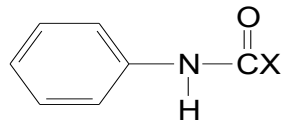
FTIR –spectrum of dye 1c

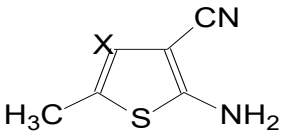


FTIR –spectrum of dye 1d

Table 6. Gas chromatography mass spectroscopy of synthesized intermediate

Sample	Fragmentation (m/z)
Calculated value	257g/mol
Intermediate	$(C_7H_6NO)^+$ =120 but m/z = 123± 3
	$(C_6H_5N_2S)^+$=137 but m/z =140±3





The gas chromatography mass spectrophotometry of the synthesize dye intermediate.

This elucidate structural information of the synthesized intermediate molecular ion of is shown in Table 6; which underwent fragmentations to produce peaks at 120g/mol but m/z is 123±3 and 137g/mol but m/z is 140±3 corresponding positive charge fragment are $C_6H_7NO^+$ and $C_6H_5N_2S^+$ respectively and its molecular mass is (257g/mol). The difference between the experimental (263 g/mol) and the calculated value (257g/mol). May be due to isotopic relative abundance ± 6.The slight difference in numerical value of the molecular weight of the) intermediate can be attributed to certain elements with high isotopic abundance [10]

Table 7. Percentage exhaustion of the dyes on nylon 6,6 and chrome tanned leather substrate

Dyes no	% E for Nylon 6,6	%fixation on nylon 6,6	%efficiency on nylon 6,6	% E CTL
1a	80.10	-	-	63.10
1b	78.50	-	-	68.50
1c	84.00	-	-	75.30
1d	87.20	-	-	68.20

Percentage exhaustion on nylon 6, 6 and exhaustion of chrome tanned leather substrates

The percentage exhaustion and fixation reported according to [10] of 2% (o.w.f) dyeing of nylon 6.6 and percentage exhaustion on chrome tanned leather substrate is summarized in Table 7. Dye 1a –1d which is monoazo acid dyes is obtained by coupling diazonium salt (2–aminothiophene) with coupling component such as 2–naphthol–3–6-disufonic acid, 8-amino-1-naphthol-3-6 disulfuric acid, 2-amino–8–naphtholsulphonic acid and 2-amino–5–naphthol–7–sulphonic acids. Indicates higher percentage exhaustion values on nylon 6,6 ranges from 78–87 while the percentage exhaustion on chrome tanned leather ranges 75–63% in which dye 1d show maximum exhaustion and dye 1c, 1a, and 1b shows minimum percentage exhaustion values 84, 80, 78% respectively. While dye 1c show maximum percentage exhaustion value 75% and dye 1b, 1d, and 1a shows minimum percentage exhaustion values 68, 68 and 63% respectively on chrome tanned leather respectively. Which indicate high levelness, penetration, substantivity and affinity due to solubility and high rate of diffusion of dye molecules.

Table 8. Light and wash fastness properties of chrome tanned dyed leather

Dyed no	Light fastness rating	Wash fastness rating
1a	5-6	4-4
1b	5	4-5
1c	5-6	4-5
1d	3-4	3-4

Table 9. Light and wash fastness properties of nylon 6,6 fabric.

Dyes	Light fastness rating	Wash fastness rating	Staining on of adjacent undyed fabric
1a	6	5	5
1b	6	5	4
1c	6	5	4
1d	7	5	5

Light fastness

Light fastness rating of the dye fabric is shown in Table 8 and 9. The test is carried out by exposing each dyed sample to day light alongside eight blue wool standards. It was observe that light fastness of dye 1a, 1b, 1c and 1d which is obtained by coupling diazonium salt with 2-naphthol-3-6 difulfonic acid, 8-amino-1-naphthol-3-6 disulfonic acid, 2-amino-8-naphthol sulfonic acid and 2-amino-5-naphthol-7-sulfonic acid to obtain monoazo acid dyes gave a light fastness rating (3-5) moderate, good and very good on chrome tanned leather, while light fastness rating (6-7) very good and excellent on nylon 6,6 substrate.

Washing Fastness

Tables 8 and 9 shows the wash fastness of the dyes coupled with diazoniun salt, 2-naphthol, 3-6 disulfonic acid, 8–amino-1-naphthol-3-6-disulfonic acid, 2-amino-8-naphthol sulphonic acid and 2-amino -5- naphthol -7- sulphonic acid. These dyes gave purple, brown, pink , deep blue to khaki hues with brighter and deeper shades, high tinctorial strength and excellent levelness on the substrates ranging from good (3), very good (4),to excellent (5) Dye 1a-1d gave a wash fastness rating on chrome tanned leather (3-5) good, very good and excellent, while on nylon 6,6 gave a wash fastness rating (5) excellent. It could be seen that nylon 6,6 gave better wash fastness than chrome tanned leather which could be as result of the texture of the fabric which in nylon 6,6 involved high diffusion and penetration and in chrome tanned leather which is interweave involve slow diffusion and penetration.

Table 10. Rubbing fastness properties on chrome tanned dyed leather

Dyed no	number of rubs	Fastness rating	Staining of adjacent un-dyed cotton
1a	100	5	4
1b	100	5	4
1c	100	5	3
1d	100	5	3

Table 11. Rubbing fastness properties of dyed nylon 6.6 fabric.

Dye No	Change in colour	Staining		Staining
1a	5	5	5	5
1b	5	5	5	5
1c	5	5	5	5
1d	5	5	5	5

Colour fastness to rubbing of the synthesized dyes on chrome tanned leather and nylon 6,6 substrates.

Rubbing fastness was designed to determine the degree of colour which may transfer from the surface of a colour fabric to a specific test substrate for rubbing. Rubbing fastness of all the dyes was very high ranging from (4-5) and the staining rating of adjacent fabric was on both nylon 6,6 and chrome tanned leather (3-4). The results obtained from the rubbing fastness test of the dyed fabric is given in Table 11: All the dyed sample gave a fastness rating (4-5) very good to excellent results. This may be due to solubilisation of a part the dye and its migration to the surface of coloured fabric [12] substantivity, penetration, levelness and durability in use.

Conclusions

The present study successfully synthesized a series of monoazo dyes based on 2-aminothiophene derivatives in moderate yields. The synthesized dyes gave a percentage yield ranging from 62-82, melting point 120-214°Cand molecular weight 507-587 g/mol. The dyes were characterized by analytical technique such as FTIR, UV-visible and GCMS of intermediate. The synthesized dyes gave a maximum absorption within the UV- visible region with λ_{max}530-597 nm which indicate bathochromic effect and molar extinction coefficient ranging from (3.217-4.221 x 10^4 l/mol^{-1} cm^{-1}) and the infra-red spectra showed peaks at 1582-1421cm^{-1} (due to azo group (N=N) stretching vibration), at 3400 – 3200 cm^{-1} due to (OH&NH) medium & broad stretching vibration), at 2830-26-95cm^{-1} (due to aromatic group (C-H) stretching vibration) , at 1760-1640 cm^{-1} (due to carbonyl group (C=O) stretching vibration), at 1342-1266 cm^{-1} (due to aromatic amine group (C-N) stretching vibration) and at 2260-2222 cm^{-1} (due to nitrile (C≡N) stretching vibration) .The dyes were found to give shade of red dark blue and darkmaroon. The dye exhibited good depth, affinity, levelling and brightness properties on the substrates. The dye gave a good percentage exhaustion on nylon 6.6 and on chrome tanned leather substrates ranging from (78-87%) and (63-75%) respectively. The dyes displayed excellent affinity towards chrome tanned leather and nylon 6,6 substrate producing vibrant shade ranging from red, dark blue and maroon . Fastness assessments indicates good to excellent wash, rubbing and light fastness properties particularly on nylon fabrics. These findings demonstrates that the synthesized dyes possess desirable dyeing potential and durability, highlighting their performance for commercial applications in textile and leather industries. Constant efforts should be made in future to study fastness properties such as perspiration, better hydrothermal resistance, hot pressing etc and effect of time and temperature on monoazo reactive dyes derived from aminothiophene.

Author Contributions

IYM: Was responsible for- Conceptualization, Methodology, Investigation, Formal analysis, Data curation, validation, visualization, Writing-Original Draft, AOA, BH, ADO, IOA: Were responsible for Writing-Review and Editing. All authors have read and approved the final version of the paper and its submission and have given consent for publication.

Declaration of Generative AI and AI-Assisted Technologies in Scientific Writing

Nothing to disclose.

Acknowledgements

None

Data Availability Statement

All relevant data are available within the manuscript

Funding
None

Conflict of Interest
The authors declare that they have no conflicts of interest.

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