Synthesis and Molecular Characterization of Diazonium Compounds Through the Reaction of Different Phenolic Compounds and Novel Aminothiophene Precursors as a New Class of Colorants

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Editor’s note: Jalali et al. developed novel diazonium compounds from two new 2-aminothiophene precursors, which have the potential to serve as a new class of colorants in medical, cosmetic, and food industries.

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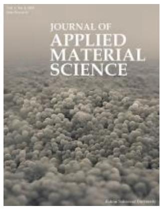

Original Research

Synthesis and Molecular Characterization of Diazonium Compounds Through the Reaction of Different Phenolic Compounds and Novel Aminothiophene Precursors as a New Class of Colorants

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Abstract

Two novel 2-amino thiophene precursors were synthesized from cyclopentanone (or cyclohexanone), sulfur, morpholine, and malononitrile using the Gewald three-component reaction. The synthesis yields of these precursors were 97% and 90%, respectively. The resulting 2-aminothiophenes were then converted into diazonium salts, which reacted in situ with various phenolic compounds, including phenol, resorcinol, and α-naphthol, to produce twelve new azo products. The novel 2-aminothiophene-based azo compounds were synthesized with yields ranging from 58% to 72% for the first set, and 54% to 72% for the second set. Subsequently, the characterization of prepared compounds was performed using ^{1}H-NMR, FT-IR, UV/Visible spectroscopy, and elemental analysis. UV/Visible spectra confirmed the presence of the azo group (N=N) with absorption in the range of 362–510 nm, which is higher than the previously reported range of 350–370 nm. The increase in λ_{max} can be attributed to the existence of an auxochrome group in the molecular structure of the compounds. Additionally, all of the various analyses performed successfully supported the synthesis of the desired compounds.

Keywords: Synthesis; Azo compounds; 2-aminothiophene; Diazonium salt; Phenolic compounds.

1. Introduction

Azo compounds are chemical compounds that contain the functional group R-N=N-R', where R and R' can be either aryl or alkyl groups [1]. These compounds have a wide range of applications, including studies in molecular biology. One notable study involved the synthesis of an aryl diazonium compound known as Dz-PEG. Dz-PEG was used for the in-situ conversion of

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serotonin into an azo compound. This conversion helps restore biotinylation efficiency by consuming serotonin through an azo-coupling reaction. Our performed proteomics analysis confirmed that biotinylation was inhibited using serotonin and that the inhibiting process was effectively reversed by Dz-PEG. [2].

A recent study presents a sensitive and rapid spectrophotometric procedure for quantifying Ketotifen fumarate in aqueous solutions. The process involves diazotizing 4-amino antipyrine using sodium nitrate in an acidic medium, then coupling it with Ketotifen to produce an orange azo dye. The dye exhibits a peak absorbance at 483 nm and remains stable for over 60 minutes. The method demonstrates high accuracy, with results ranging from 98% to 100.16%, making it practical for pharmaceutical formulations. Compared to existing approaches, it provides notable advantages [3-6], offering superior sensitivity and better solvent suitability [7].

Azo dyes are commonly used as colorants in the textile industry [8, 9]. They are also used as pigments in digital printing [10]. These dyes are utilized in medicine and the cosmetic and food industries [11]. They are valuable in the preparation of advanced materials, too [12]. Some dyes are used in the biomedical field [13], exhibiting antibacterial [14] and Antifungal Activity [15]. From their other applications, we can point to their usage as chiral receptors [16], liquid crystals [17], new glassy materials [18], and chiral switches in photochemistry [19]. They have been applied as semiconductors and optical data storage Equipment [20].

Aminothiophene-based azo dyes are recognized as dyes that exhibit great brightness. They have emerged as a cost-effective alternative to more expensive anthraquinone dyes [8]. The preparation of novel azo derivatives from insoluble amines in aqueous media is crucial as it could lead to technological advancements. In this paper, we report the preparation of several novel azo compounds through a coupling reaction of 2-aminothiophene with phenolic compounds.

2. Experimental

2.1. Materials

Malononitrile, sulfur, cyclohexanone, cyclopentanone, morpholine, HCl (98%), NaOH, resorcinol, catechol, phloroglucinol, phenol, α-naphthol, and β-naphthol were purchased from Merck and were of analytical grade. All chemicals were used as received without further purification, and solvents were redistilled before use.

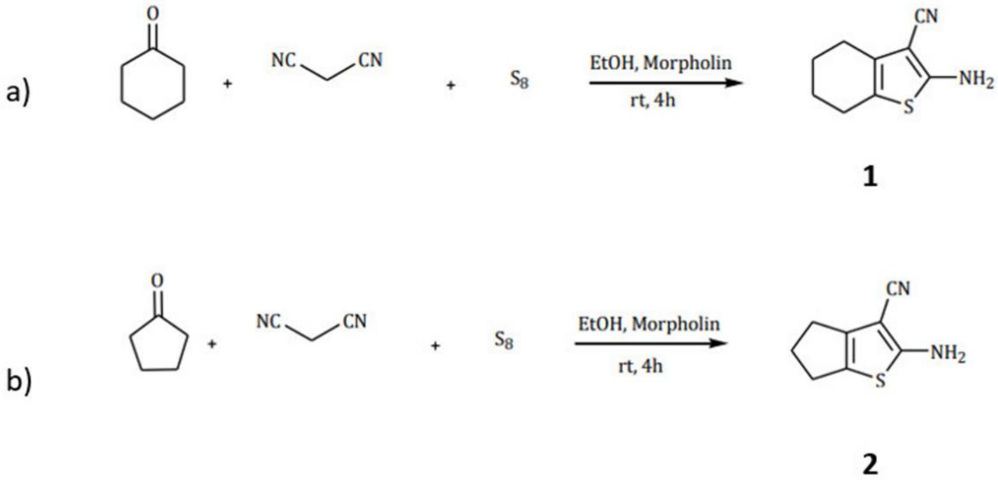
2.2. Synthesis of 2-amino-4,5,6,7-tetrahydro-benzo[b]thiophene-3-carbonitrile

First, 132 grams (2 moles) of malononitrile, 64 grams (2 moles) of sulfur, and 800 mL of ethanol at 20°C, 200 mL (2.3 moles) of morpholine were slowly added to a mixture of 200 grams (2 moles) of cyclohexanone. The mixture was then stirred for four hours, and the formed precipitate was filtered using a Buchner funnel and then washed with distilled water and ethanol, as described in the literature [21]. It was subsequently recrystallized in hot ethanol, resulting in the final compound, which appeared as cream-colored needle crystals with a yield of 97%.

As illustrated in Scheme 1, the first step of this reaction involves the Neunagel-Koop condensation between an active nitrile and a ketone or aldehyde, leading to the production of acrylonitrile. The product of this condensation undergoes sulfurization at the methylene position, followed by a ring addition reaction between the mercaptide and the cyano group. After the alkali catalysis step, the catalyst is removed, resulting in the formation of 2-aminothiophene. The melting point of compound (1) is 145°C, and the distinct TLC spots compared to the starting materials indicate the successful formation of this compound [33]. The IR spectrum data for this compound indicates a peak at 2192 cm^{-1}, associated with the C-N group. Additionally, the peaks observed in the range of 3442-3325 cm^{-1} are associated with the NH_2 group of the primary amine. A peak at 1618 cm^{-1} corresponds to the C=C bond, and the peak at 2914 cm^{-1} is related to the stretching frequency of the aliphatic C-H bond. A similar process was used to synthesize compound 2.

2.3. Synthesis of 2-amino-5,6-dihydro-4H-cyclopenta[b]thiophene-3-carbonitrile

Morpholine (200 mL, 2.3 mol) was slowly added to a mixture of cyclopentanone (200 grams, 2 mol), malononitrile (132 grams, 2 mol), sulfur (64 grams, 2 mol), and 800 mL of ethanol at 20°C. The mixture was stirred for four hours, after which the formed solid was isolated using conventional methods [21]. Then, it was



Scheme 1. Synthesis of amino thiophenes (1 and 2) using the Gewald synthesis method: (a) Reaction with cyclohexanone and (b) Reaction with cyclopentanone.

diluted with water, and the final precipitate was filtered using a Buchner funnel and washed with distilled water and ethanol. The resulting precipitate was recrystallized in hot ethanol, yielding the final compound as brown pellets with a yield of 90%.

The melting point of compound (2) is 142°C. The IR spectrum of this compound indicates the formation of the compound through the following data: the peak of the C-N group (at approximately 2192 cm^{-1}), the NH_2 group (at approximately 3326 and 3421 cm^{-1}), the C=C bond (at approximately 1614 cm^{-1}), and the C-H bond (at approximately 2910 cm^{-1}).

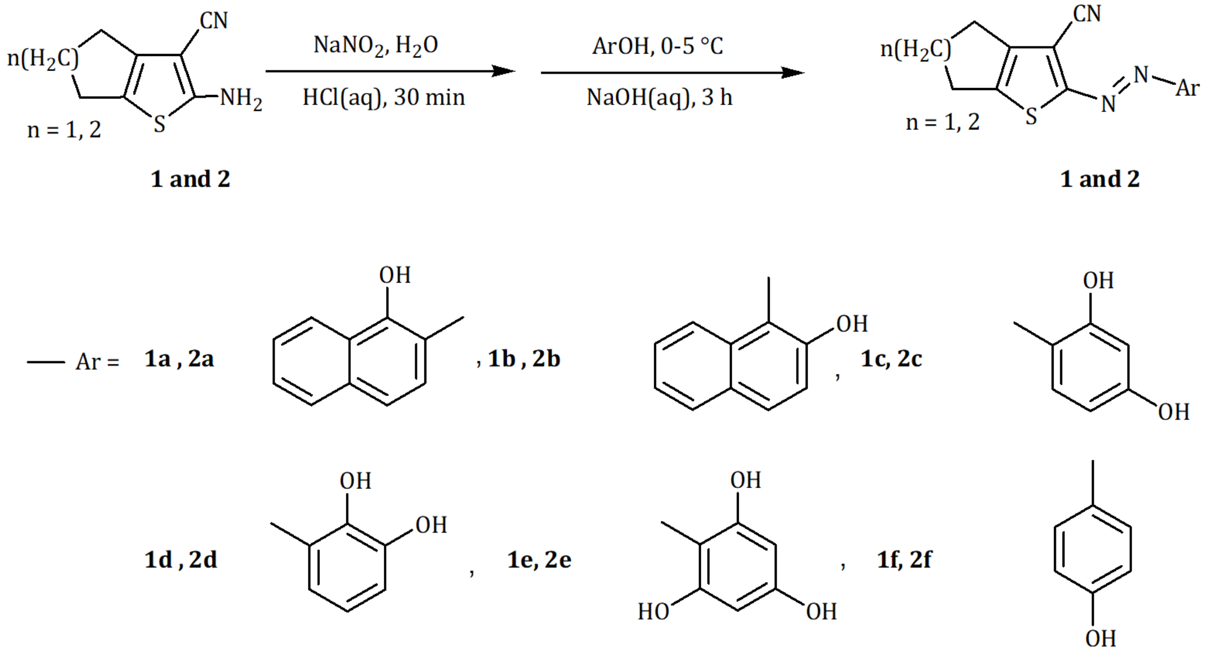
2.4. Measurements

All reactions were monitored using thin-layer chromatography (TLC) on commercially available pre-coated glass plates (silica gel 60 F_{254}, Merck), which were visualized under UV light at 254 nm. Melting points were recorded using an uncorrected Electrothermal-9200 melting point apparatus. Fourier-transform infrared (FT-IR) spectra were measured at room temperature with a Shimadzu 8400 FT-IR instrument. Proton nuclear magnetic resonance (^1H NMR) spectra were obtained using a Bruker 300 MHz NMR Spectrometer, with tetramethylsilane (TMS) as the internal reference. Ultraviolet/visible (UV/Vis) spectroscopy analyses were performed using a Shimadzu UV-1601 PC instrument.

3. Results and discussion

The synthesis of the novel azo compounds is illustrated in Scheme 2. The characterization data for the newly prepared azo compounds are summarized in Table 1. The FT-IR data (reported in Table 2) for new azo compounds exhibited characteristic bands corresponding to the coupling agents 1 and 2, the nitrile, hydroxyl, and alkene functional groups. Additionally, the existence of the (N=N) bond was confirmed by a band observed in the range of 1456–1650 cm^{-1}, indicating the success of the synthesis process. Azo compound 1a was selected as a representative example for the ^1H-NMR analysis, and the corresponding spectra are displayed in Figure 1. The ^1H-NMR spectral data for the newly prepared azo compounds are presented in Table 3.

The ^1H-NMR spectrum of compound 1a (Figure 1) indicates that the hydrogen of the O-H group appears at a higher chemical shift than the other prepared derivatives. This phenomenon is likely due to the proximity of the O-H group to the -N=N- double bond, which may result in tautomerism and the replacement of the hydrogen atom in the O-H group (Scheme 3). Based on the ^1H-NMR spectrum of this compound, the peak corresponding to the N-H amino group appears as a singlet at approximately 13.5 ppm. It can be attributed to the formation of an intramolecular hydrogen bond and the highly acidic nature of this hydrogen.



Scheme 2. Synthetic details of twelve novel azo compounds introduced in this work.

The hydrogen attached to carbon number 6, which is in the ortho position relative to the carbonyl group, is observed as a doublet around 7.8 ppm. This doublet results from the electron-withdrawing effect of the C=O group. The hydrogen bonded to carbon atom number 1 appears as a doublet peak in the region of 8.6 ppm. The hydrogens bonded to carbon atoms 2 and 3 appear as two triplet peaks at 7.4 ppm and 7.5 ppm, respectively. Hydrogen 4 is observed as a doublet peak at 7.7 ppm. Hydrogen 5, which is situated in the meta position relative to the carbonyl group, appears as a doublet peak at 7.2 ppm. The hydrogens attached to carbon atoms 9 and 10, which share the same chemical environment, appear as a singlet peak at 1.8 ppm. Finally, the hydrogens attached to carbon atoms 8 and 11 appear as two singlet peaks at 2.7 ppm and 2.8 ppm, respectively.

The FT-IR spectra (Figure 2) of this compound (1a) include: stretching frequency related to the OH group

Table 1. Characterization results of synthesized azo compounds

Compound	Formula	Molar Mass (g/mol)	Weight Fraction, calc. (%)					Yield (%)	Melting Point (°C)
			C	H	N	S	O		
1	$C_9H_{10}N_2S$	178.26	53.89	5.65	15.71	17.99	0	97	145
2	$C_8H_8N_2S$	164.23	58.50	4.9	17.06	15.52	0	90	142
1a	$C_{19}H_{15}N_3SO$	333.41	68.44	4.53	12.60	9.61	4.79	72	260
1b	$C_{19}H_{15}N_3SO$	333.41	68.44	4.53	12.60	9.61	4.79	59	220
1c	$C_{15}H_{13}N_3SO_2$	299.35	60.18	4.37	14.04	10.71	10.71	81	243
1d	$C_{15}H_{13}N_3SO_2$	299.35	60.18	4.37	14.04	10.71	10.71	79	256
1e	$C_{15}H_{13}N_3SO_3$	315.35	57.12	4.15	13.32	10.16	15.22	67	278
1f	$C_{15}H_{13}N_3SO$	283.35	63.57	4.62	14.83	11.31	5.64	58	234
2b	$C_{18}H_{13}N_3SO$	319.38	67.68	4.10	13.15	10.04	5.00	54	206
2c	$C_{15}H_{13}N_3SO_2$	283.35	63.57	4.62	14.83	11.31	11.29	71	236
2d	$C_{15}H_{13}N_3SO_2$	283.35	63.57	4.62	14.83	11.31	11.29	68	245
2e	$C_{15}H_{13}N_3SO_3$	315.35	57.12	4.15	13.32	10.16	15.22	72	267
2f	$C_{15}H_{13}N_3SO$	283.35	63.57	4.62	14.83	11.31	5.64	62	228

Table 2. The FT-IR spectral data of newly prepared azo compounds

Compound	Spectral data
1	IR, v/cm^{-1}: 1247 (C-N), 1417 (C=C), 1456 (N=N), 2214 (C≡N), 3064 (CH), 3346 (OH)
2	IR, v/cm^{-1}: 1303 (C-N), 1523 (C=C), 1566 (N=N), 2223 (C≡N), 2927 (CH), 3238 (OH)
1a	IR, v/cm^{-1}: 1319 (C-N), 1458 (C=C), 1620 (N=N), 2221 (C≡N), 3072 (CH), 33303 (OH)
1b	IR, v/cm^{-1}: 1442 (C-N), 1523 (C=C), 1562 (N=N), 2214 (C≡N), 2928 (CH), 3423 (OH)
1c	IR, v/cm^{-1}: 1427 (C-N), 1521 (C=C), 1639 (N=N), 2225 (C≡N), 2935 (CH), 3334 (OH)
1d	IR, v/cm^{-1}: 1434 (C-N), 1515 (C=C), 1641 (N=N), 2225 (C≡N), 2937 (CH), 3400 (OH)
1e	IR, v/cm^{-1}: 1303 (C-N), 1508 (C=C), 1436 (N=N), 2356 (C≡N), 3218 (CH), 3438 (OH)
1f	IR, v/cm^{-1}: 1334 (C-N), 1515 (C=C), 1569 (N=N), 2206 (C≡N), 3421 (CH), 3226 (OH)
2b	IR, v/cm^{-1}: 1340 (C-N), 1440 (C=C), 1566 (N=N), 2227 (C≡N), 3049 (CH), 3232 (OH)
2c	IR, v/cm^{-1}: 1434 (C-N), 1581 (C=C), 1620 (N=N), 2202 (C≡N), 3055 (CH), 3197 (OH)
2d	IR, v/cm^{-1}: 1407 (C-N), 1521 (C=C), 1631 (N=N), 2202 (C≡N), 2920 (CH), 3413 (OH)
2e	IR, v/cm^{-1}: 1434 (C-N), 1515 (C=C), 1641 (N=N), 2225 (C≡N), 2937 (CH), 3400 (OH)
2f	IR, v/cm^{-1}: 1247 (C-N), 1417 (C=C), 1456 (N=N), 2214 (C≡N), 3064 (CH), 3346 (OH)

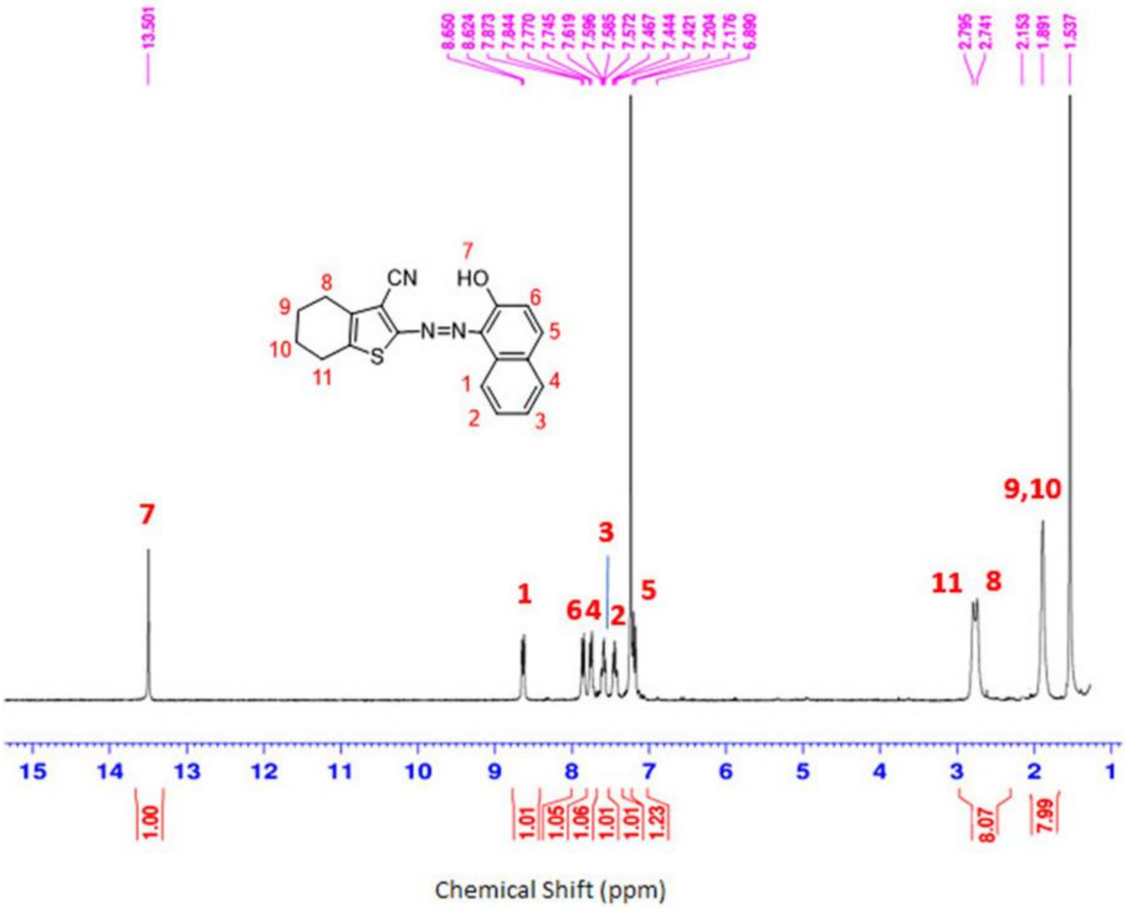


Figure 1. ^{1}H-NMR spectrum of 1**a**.

Table 3. ^{1}H-NMR spectral data of newly prepared azo compounds

Compound	**Spectral data**
1a	^{1}H NMR ($CDCl_3$), δ/ppm: 1.8 (s, 4H, CH_2), 2.7 (s, 2H, CH_2), 2.8 (s, 2H, CH_2), 6.8 (d, *J*=8.4 Hz , 2H, ArH), 6.8 (d, *J*=8.4 Hz, 2H, ArH), 7.4 (t, *J*=6.9 Hz, 1H, ArH), 7.5 (t, *J*=14.01 Hz, 1H, ArH), 7.7 (d, J=7.5 Hz, 1H, ArH), 7.8 (d, J=8.7 Hz, 1H, ArH), 8.6 (d, *J*=7.8 Hz, 1H, ArH), 13.5 (s, 1H, OH).
1b	^{1}H NMR (DMSO-d_6), δ/ppm: 2.3 (s, 4H, CH_2), 2.7 (s, 2H, CH_2), 2.9 (s, 2H, CH_2), 7.1 (d, *J*= 7.8 Hz, 1H, ArH), 7.2 (m, 2H, ArH), 7.9 (d, *J*= 7.8 Hz, 1H, ArH), 8.2 (d, *J*= 7.8 Hz, 1H, ArH), 8.63 (d, *J*= 7.8 Hz, 1H, ArH), 11.63 (s, 1H, OH).
1c	^{1}H NMR (DMSO-d_6), δ/ppm: 1.8 (s, 4H, CH_2), 2.6 (s, 2H, ArH), 2.7 (s, 2H, ArH), 6.4 (s, 1H, ArH), 6.5 (d, *J*= 3 Hz, 1H, ArH), 7.83 (d, *J*= 1.2 Hz, 1H, ArH), 10.88 (s, 1H, OH), 11.08 (s, 1H, OH).

Notes: DMSO: dimethyl sulfoxide, *J*: coupling constant

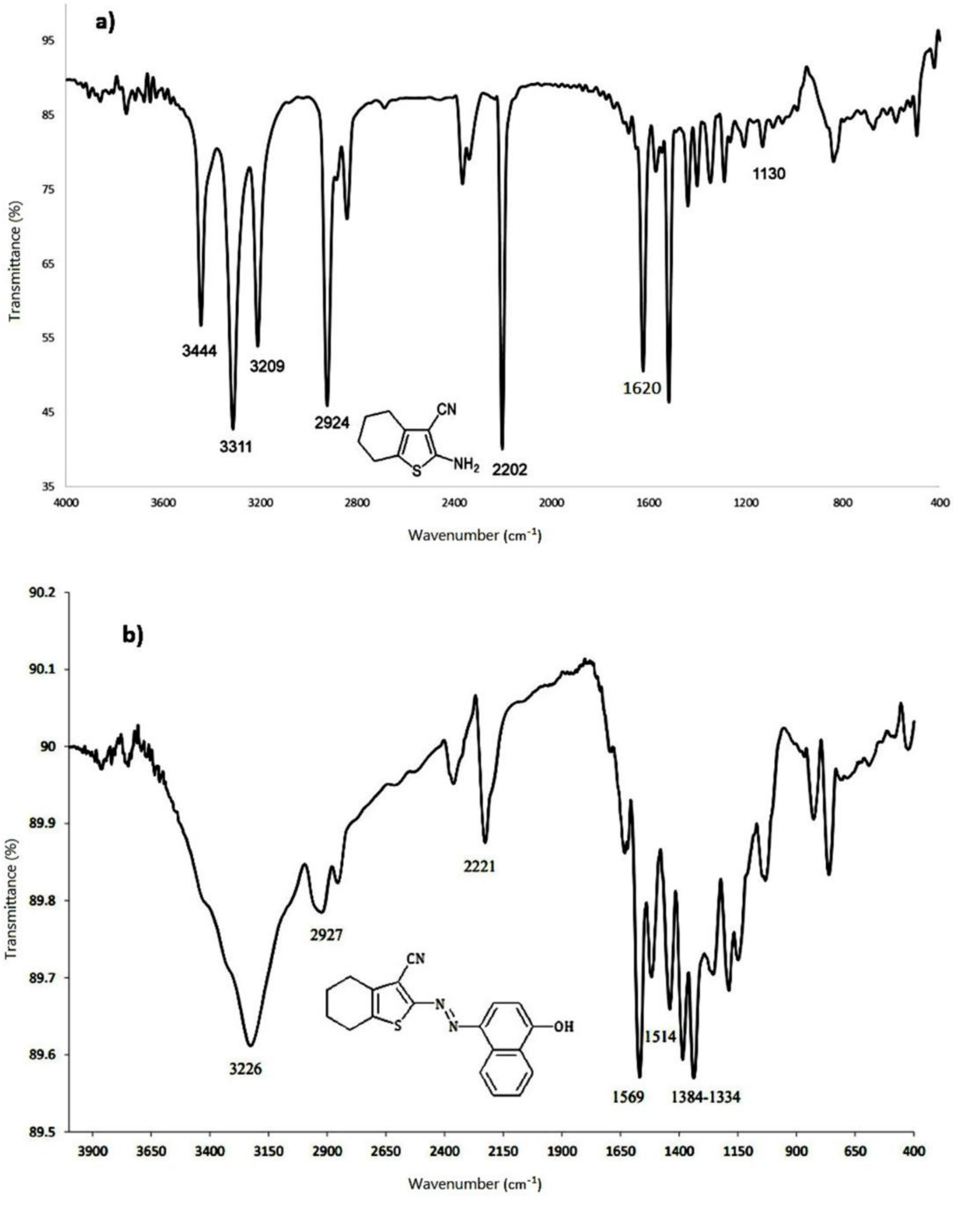
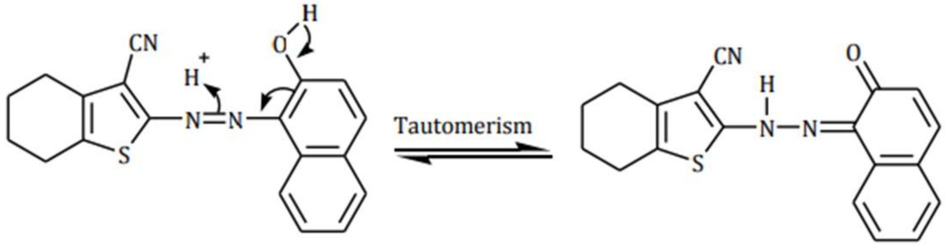


Figure 2. FT-IR spectra of: (a) **1** and (b) **1a**.



Scheme 3. The tautomerization in compound 1a.

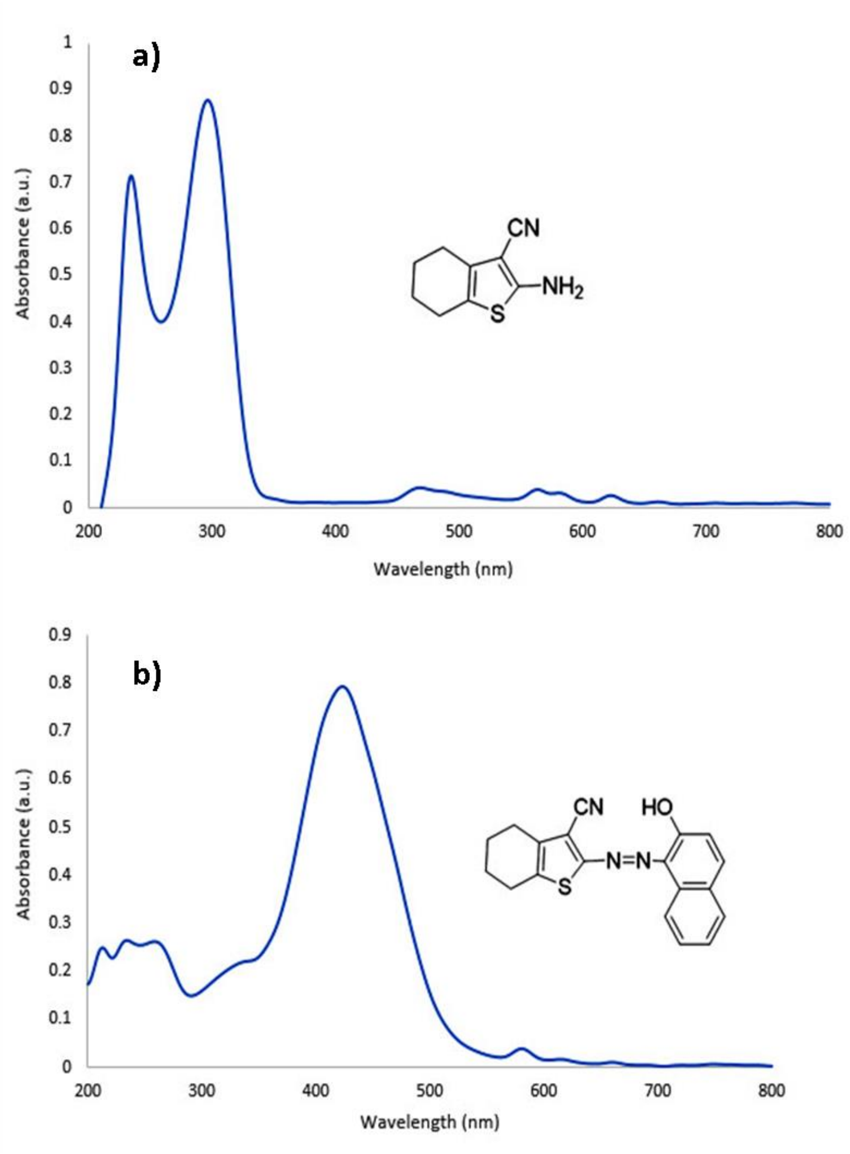
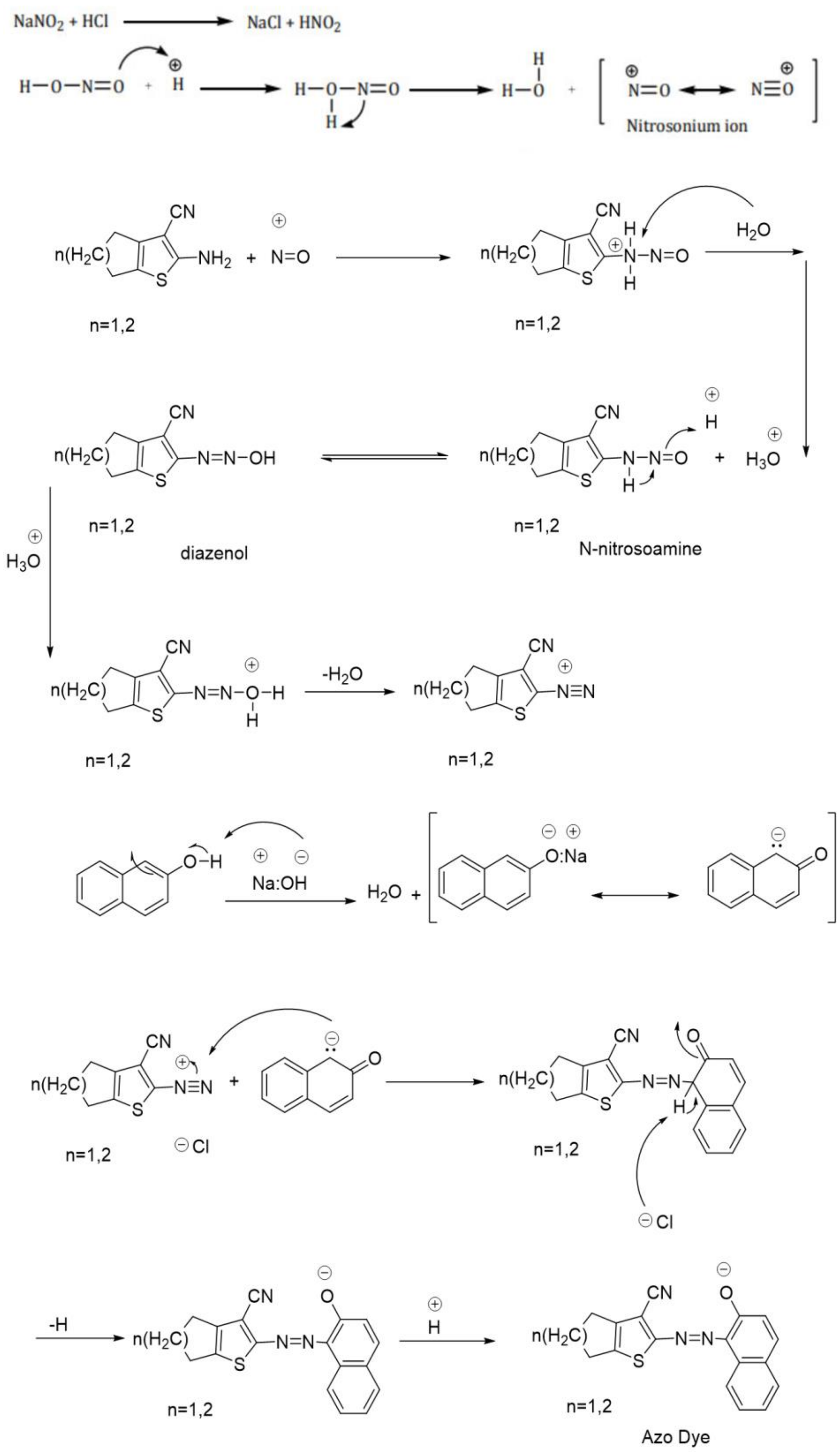


Figure 3. UV-vis spectra of: (a) **1** and (b) **1a**.



Scheme 4. The mechanism of coupling of aminothiophenes (1 and 2) with 2-naphthol.

Table 4. UV-vis spectral data of newly prepared azo compounds

Compound	Spectral data of UV-vis: λ_{max} (nm)	Color of the compound
1	UV-vis (DMSO): λ_{max} 510 nm	dark red, almost brown
2	UV-vis (Acetone): λ_{max} 507 nm	dark red
1a	UV-vis (Acetone): λ_{max} 470 nm	light brown
1b	UV-vis (Acetone): λ_{max} 469 nm	brown
1c	UV-vis (Acetone): λ_{max} 477 nm	light red
1d	UV-vis (Acetone): λ_{max} 368 nm	brown
1e	UV-vis (DMSO): λ_{max} 506 nm	dark red
1f	UV-vis (Acetone): λ_{max} 499 nm	dark red, almost black
2b	UV-vis (Acetone): λ_{max} 468 nm	dark brown
2c	UV-vis (Acetone): λ_{max} 467 nm	brown
2d	UV-vis (Acetone): λ_{max} 475 nm	dark red
2e	UV-vis (Acetone): λ_{max} 362 nm	dark brown
2f	UV-vis (DMSO): λ_{max} 510 nm	dark red, almost brown

(due to the formation of an intramolecular hydrogen bond) at around 3346 cm^{-1}, the aromatic peak of C-H is in the area of 3064 cm^{-1}. The peak related to the N=N bond is in the region of 1456 cm^{-1}. A peak of aromatic C=C bond appears at 1417 cm^{-1}. The peaks related to the C≡N bond and C-N bond are observed at 2214 and 1247 cm^{-1}, respectively. As expected, the FT-IR spectra of **1** and **1a** exhibited the same characteristic bands of the coupling agent **1,** namely nitrile and alkene groups. In contrast, the existence of the azo band in 1456 cm^{-1} and hydroxyl group bond in 3346 cm^{-1} [22] in the FT-IR spectrum of **1a**, confirmed that the synthesis is successful. The proposed reaction mechanism appears in Scheme 4.

Figure 4. Digital images of samples: (a) 1 and (b) 1a.

The azo group (N=N) usually gives an absorption in the 350–370 nm range [23]. As expected, the examination of Figure 3 showed that the new azo compounds (1a, for example) would exhibit higher λ_{max} due to the presence of the hydroxyl group in the compounds [24]. The results indicated a positive outcome, revealing a higher absorption wavelength ranging from 362 to 510 nm. The UV/vis data for the azo compounds 1a-f and 2a-f are presented in Table 4. Additionally, images of samples 1 and 1a are shown in Figure 4.

4. Conclusions

In summary, two new 2-aminothiophene compounds were synthesized with excellent yields using the Gewald three-component reaction. Additionally, twelve new azo compounds based on 2-amino-4, 5, 6, 7-tetrahydrobenzo[b]thiophene-3-carbonitrile and 2-amino-5, 6-dihydro-4H-cyclopenta[b]thiophene-3-carbonitrile were successfully synthesized with good yields. These compounds were characterized using ^{1}H-NMR, FT-IR, UV/Visible spectroscopy, and elemental analysis. All analyses confirmed the successful synthesis of the desired products.

Conflict of Interest

The authors declare no conflict of interest.

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