

PTSA CATALYZED SYNTHESIS OF SCHIFF'S BASE VIA 1, 3, 4-THIADIAZOL-2-AMINE

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ABSTRACT

An effective technique for creating a new Schiff base (5a-5f), (E)-5-styryl-1, 3, 4-thiadiazol-2-amine, with P-substituted aromatic aldehyde in the presence of P-toluenesulphonic acid at room temperature in ethanol as a solvent and thiosemicarbazide can be converted into the intermediate molecule (E)-5-styryl-1, 3, 4-thiadiazol-2-amine (3) in the presence of an acid medium. Advanced spectroscopic data (^1H NMR, ^{13}C NMR, and LCMS) were used to analyze all of the freshly synthesized derivatives. Additionally, elemental analysis was used to determine the structural determination of the compounds. All of the newly discovered compounds were then examined for their antibacterial properties.

KEYWORDS: (E)-5-styryl-1,3,4-thiadiazol-2-amine, substituted aromatic aldehydes, P-toluene sulfonic acid, Schiff bases, bioevaluation.

1. INTRODUCTION

First described by Hugo Schiff, Schiff bases are imine or azomethine ($-\text{C}=\text{N}-$) functionalities that are primarily synthetic results of the condensation process between primary amines and active aromatic carbonyl moieties using appropriate solvents. Schiff bases' reported diverse pharmacological properties, which include antimicrobial Biological Studies,^[1,2] Antimicrobial Activities,^[3-8] Anticancer,^[9] Antioxidant Activity,^[10,11] antiviral,^[12-13] analgesic and anti-inflammatory activity,^[14] DNA topoisomerase I and Hela, MCF7 and A431 cells,^[15,16] Antitumor Activity,^[17] clearly demonstrate their medicinal potential in drug design. Their improved lipophilic nature, which increases their bioavailability in lipid membranes, may be the cause of their increased reported bioactivity.

The SBs and their mineral derivatives can be made by a variety of common methods, including the following: oxidation synthesis of amines from alcohols and amines, condensation of aldehydes, cyanide addition to organometallic reagents, the reaction of phenols/phenol ethers with nitriles, the metal amides reaction, the preparation of ketamine using ketals, the reaction of hydrazoic acid with tertiary alcohols and olefins, the conversion of alpha-amino acids to imines, and the reduction of nitro-compounds.

The type of nucleophiles (Amines) that are added to the carbonyl group determines the SB's production mechanism. An unstable substance called carbinolamine is produced when the amine first interacts with the aldehyde or ketone. Acid and/or base-catalyzed pathways cause this molecule to lose water. Because carbinolamine is an alcohol, it can become dehydrated when exposed to acid.

The Schiff's bases have received considerable interest and several methodologies for their synthesis have been reported. This involves the use of acidic catalysts such as Methane Sulfonic Acid^[18] Heterogeneous Acid,^[19] cerium (IV) ammonium nitrate (CAN),^[20] P2O5/SiO2,^[21] Mild Steel as an Acidic Corrosion Inhibitor^[22] solvent free conditions utilizing catalytic amounts of p-TSA under conventional method .

In this study, we used a methane sulphonic acid catalyst to create Schiff base from (E)-5-styryl-1,3,4-thiadiazol-2-amine(3) and several P-substituted aryl aldehydes (One that donates electrons, one that withdraws electrons, and one that contains halogen). We set out to create new Schiff's bases using the organic acid P-toluene sulphonic acid catalyst because it produces a higher yield, takes less time to complete, and allows for the synthesis of cinnamic acid with semithiocarbazide as an intermediate in the reaction, such as the molecule (E)-5-styryl-1, 3, 4-thiadiazol-2-amine. Moreover, the biological activity was examined. Considering these facts, a continuous search for Schiff base's antibacterial action is underway.

2. MATERIALS AND METHODS

The chemicals reagents, solvents were used for this synthesis were purchased from Sigma Aldrich. The melting point was determined with a melting point apparatus and was uncorrected. Thin Layer Chromatography (TLC) was carried out using a Merck pre-coated silica gel plate (10 x 10 cm), the R_f value obtained using ethyl acetate as the mobile phase and the spot located and visualized using an ultraviolet lamp at 256 nm. ¹H NMR and ¹³C

NMR spectrum of the sample was recorded on a JEOL Eclipse 400 NMR spectrophotometer by JEOL (Pleasanton, USA) using DMSO-d₆.

2.1. Preparation of (E)-5-Styryl-1,3,4-Thiadiazol-2-amine(3)

Take dry and clean four necks RBF. The mixture of cinnamic acid (1mol) and semithiocarbazide (1.25mol) dissolved in the toluene (25mL) and few drops of phosphoric acid (H₂SO₄) into RBF at room temperature which is also fitted on the magnetic stirrer containing hot plate. The reaction mixture continuous carried the reaction for 5 hrs. at 100°C. The progress of the reaction checked by the TLC (EtOAc: n-hexane = 5:5). After all the reactants were consumed and then cooled the reaction mixture at RT. The crude dissolved in ethyl acetate and washed with as saturated solution of sodium bicarbonate and separated the ethyl acetate layer and also washed with water separated the organic layer. The organic layer can be distilled off under vacuums and solid compound obtained.

Yellow compound, yield-89%, M.P – 141-143°C, ¹H NMR (400 MHz, CDCl₃) δ ppm: 7.614–7.324(m, 5, Ar-H), 7.025(s, 2H, NH₂), 6.451(s, 1H, =CH), 6.652(s, 1H, =CH); ¹³C NMR (100MHz, CDCl₃) δ ppm: 168.25, 154.22, 135.36, 128.54, 128.12, 127.96, 1; Molecular weight(m/z); C₁₀H₉N₃S; Molecular formulae : 204.35(M+H). Elemental analysis: Calculated: C-59.09, H-4.46, N-20.67; Found: C-59.02, H-4.45, N-20.76.

2.2. General procedure for the synthesis of schiff base(5a-5f)

A mixture of equimolar quantities (1m mol) of (E)-5-styryl-1,3,4-thiadiazol-2-amine and substituted aromatic carbaldehyde (1.2m mol) was dissolved in 20 ml of dry ethanol taken in RB flask and subsequently added catalytic amount P-Toluene sulphonic acid (4% mmol) to be mixture. The reaction mixture carried out under RT condition 4 h. The reaction was monitored by TLC. The mixture of the compound with extracted with ethyl acetate and washed with and solution of sodium bicarbonate. Finally product can be obtained by after re-crystallized from ethanol.

2.2.1. 1-phenyl-N-(5-((E)-styryl)-1,3,4-thiadiazol-2-yl)methanimine(5a)

Pale yellow compound, yield-86%, m.p – 212-214°C, ¹H NMR (400 MHz, CDCl₃) δ ppm: 8.465(=CH, S, 1H), 7.810–7.236(m, 10H, Ar-H), 6.536(s, 1H, =CH), 6.137(s, 1H, =CH); ¹³C NMR (100MHz, CDCl₃) δ ppm: 168.74, 155.06, 135.55, 133.07, 131.73, 129.98, 129.54, 128.86, 128.41, 128.19, 127.77, 117.43; Molecular weight(m/z); C₁₇H₁₃N₃S Molecular formulae :

292.31(M+H). Elemental analysis: Calculated: C-70.08, H-4.50, N-14.42; Found: C-70.02, H-4.48, N-14.48.

2.2.2. 4-(((5-((E)-styryl)-1,3,4-thiadiazol-2-yl)imino)methyl)phenol(5b)

Pale yellow compound, yield -89%, m.p - 221-223⁰C; ¹H NMR (400 MHz, CDCl₃)δ ppm :9.543 (s, 1H, 6H), 8.665 (s, 1H, =CH), 7.713- 7.326 (m, 7H, Ar-H), 6.963 – 6.812 (m, 2H, Ar-H), 6.712 (s, 1H, =CH), 6.463 (s, 1H, =CH); ¹³CNMR(100MHz, CDCl₃)δ ppm :157.29, 155.09, 150.26, 136.11, 132.96, 130.44, 129.68, 128.57, 128.33, 127.49, 127.05, 116.06; Molecular weight(m/z); C₁₇H₁₃N₃OS Molecular formulae: 308.74(M+H). Elemental analysis: Calculated: C-66.43, H-4.26, N-13.62; Found: C-66.37, H-4.32, N-13.67.

2.2.3. N-(5-((E)-styryl)-1,3,4-thiadiazol-2-yl)-1-(3,4,5-trimethoxyphenyl)methanimine(5c)

Pale yellow compound, yield - 91%, m.p -207-209⁰C; ¹H NMR (400 MHz, CDCl₃)δ ppm:8.814 (s, 1H, =CH), 7.766 – 7.314 (m, 5H, AR-H), 7.218-7.046 (m, 2H, Ar-H), 3.796(s, 6H, (OCH₃)₂), 3.665 (s, 6H, (OCH₃)₂), 6.818 (J = 7.24, 1H, d), 6.785(J =7.24, 1H, d) ¹³CNMR(100MHz, CDCl₃)δ ppm :161.72, 157.04, 150.29, 138.55, 135.22, 132.06, 130.84, 128.78, 128.32, 127.66, 127.19, 117.44, 61.83, 54.36. Molecular weight (m/z); C₂₀H₁₉N₃O₃S Molecular formulae : 382.54(M+H). Elemental analysis: calculated: C-62.98, H-5.02, N-11.02; Found: C-62.91, H-5.01, N-11.07.

2.2.4. 1-(4-chlorophenyl)-N-(5-((E)-styryl)-1,3,4-thiadiazol-2-yl)methanimine(5d)

Pale yellow compound, yield – 87%, m.p -198-200⁰C; ¹H NMR (400 MHz, CDCl₃)δ ppm :7.4188.668 (s, 1H), 7.814(d, J =8.8, 2H), 7.643 (d, J=7.6, 2H), 7.418(d, J=8.0, 2H), 7.374(d, J=6.812, 2H), 6.744(d, J=10.2, 1H); ¹³CNMR(100MHz, CDCl₃)δ ppm :159.84, 156.26, 136.07, 133.76, 132.38, 130.29, 129.92, 128.02, 128.68, 128.26, 127.19, 117.28; Molecular weight(m/z); 327.28(M+H). Molecular formulae: C₁₇H₁₂ClN₃S; Elemental analysis: calculated: C-62.67, H-3.71, N-12.90; Found : C-60.61, H-3.70, N-12.95.

2.2.5. 1-(4-bromophenyl)-N-(5-((E)-styryl)-1,3,4-thiadiazol-2-yl)methanimine(5e)

Dark yellow compound, yield – 88%, m.p -215-216⁰C, ¹H NMR (400 MHz, CDCl₃)δ ppm :8.715(s, 1H, =CH), 7.914 – 7.736(m, 2H, Ar-H), 7.517-7.296(m, 5H, Ar-H), 6.846(d, J=7.4, 1H), 6.638 (d, J=7.4, 1H); ¹³CNMR(100MHz, CDCl₃)δ ppm :162.68, 157.69, 158.05, 134.78, 132.36, 130.04, 128.76, 128.44, 128.19, 127.65, 127.16, 115.38; Molecular weight(m/z);

370.49(M+H). Molecular formulae: $C_{17}H_{12}BrN_3S$; Elemental analysis: calculated: C-55.15, H-3.27, N-11.35; Found: C-55.08, H-3.25, N-11.42

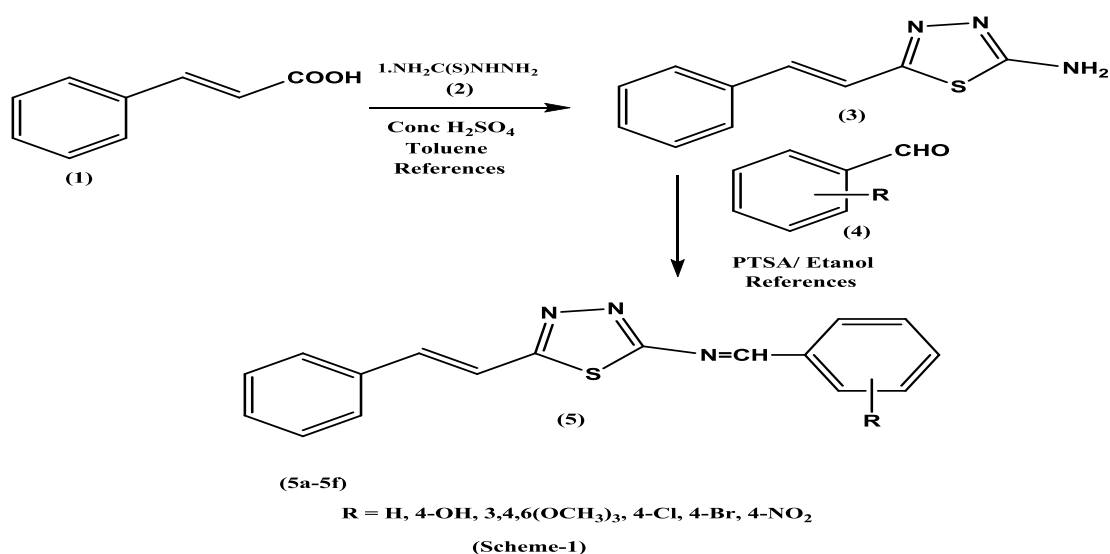
2.2.6. 1-(4-nitrophenyl)-N-(5-((E)-styryl)-1,3,4-thiadiazol-2-yl)methanimine(5f):

Pale yellow compound, yield – 82% , m.p -205-207⁰C ; ¹HNMR (400 MHz, $CDCl_3$) δ ppm: 8.896(s,1H,=CH),8.269–8.015(m,4H,Ar-H), 7.665-7.339(m,5H,Ar-H), 6.884(d, J=5.2,1H), 6.765(d,J=5.2Hz,1H);¹³CNMR(100MHz, $CDCl_3$) δ ppm:164.46,159.36,146.62,140.38, 138.15, 133.05,128.89,128.47 128.05, 127.46, 125.67,117.38;Molecular weight(m/z) ; 337.17(M+H). Molecular formulae: $C_{17}H_{12}N_4O_2S$; Elemental analysis: calculated: C-60.70, H-3.60, N-16.66; Found: C-60.63, H-3.58, N-16.71.

3. RESULTS AND DISCUSSION

Herein, the reaction condition carried out at room temperature for all the newly synthesized compounds. An effective technique for creating a new Schiff base(5a-5f), (E)-5-styryl-1, 3, 4-thiadiazol-2-amine, with P-substituted aromatic aldehyde in the presence P-toluenesulphonic acid at room temperature ethanol as a solvent and the cinnamic acid and with thiosemicarbaide can be converted into the intermediate molecule (E)-5-styryl-1, 3, 4-thiadiazol-2-amine(3) in the presence of acid medium.

The total number of sampled observation of the yield of six derivatives is not the same, therefore the difference among the effect of the derivatives to obtain by considering the following hypothesis.



These analogous reactions were optimized using different catalysts, different amounts of catalyst, and different solvents. While varying amounts of catalyst were used during the reaction below, the highest product of the derivatives was obtained in the presence of protic acid), methanesulphonic acid (MSA) catalyst compared to oxidative related catalysts such as silica supported sulphonic acid (SSA), methanesulphonic acid (MSA), toluene sulphonic acid (PTSA), camphorsulphonic acid (CSA), and trichlorosalicylic acid (TCSA) (Table I).

Table I: The effect of catalyst for preparation of titled derivatives.

Entry	Catalyst	Time (hrs.)	Yield (%)
1	SSA	4	45
2	MSA	4	54
3	PTSA	4	91
4	CSA	4	72
5	TCSA	4	67

DMF, isopropanol acetonitrile, ethanol, and methanol were among the different solvents used in the model reaction that was investigated. With an 89% product yield, it was determined to be the most effective medium for the reaction. As a result, it was utilized as the solvent for further reactions due to its higher yield, environmentally friendly nature, and ease of work-up. The solvent significant role play is ethanol and reactions due to its higher yield, environmentally friendly nature, and ease of work-up. The solvent significant role play is ethanol.

Table II: The effect of solvent for preparation of titled derivatives (5c).

Entry	Catalyst	Time (hrs.)	Yield (%)
1	DMF	3	55
2	IPA	3	63
3	CH ₃ CN	3	49
4	EtOH	3	91
5	MeOH	3	69

Table -III illustrates a notable improvement in the targeted compounds, with 5c's yield being developed to 91%. The quantity of the catalyst used in the synthesis, the impact on the product yield, and the rate of reaction. The variation of the loaded catalyst was found to improve the product, as indicated in table III. The results could not be enhanced by using the maximum amounts of the catalyst. The yield unexpectedly dropped to 30%, as indicated in Table III, even though the reaction time was reduced to 1 hour by using 4.0 mmol% CSA

Table III: The effect of loaded for preparation of titled (5c).

Entry	Amount catalyst (mmol)	Time (hrs)	Yield (%)
1	1.0	3	20
2	2.0	3	46
3	4.0	3	91
4	6.0	3	91

3.1. Antimicrobial activity

The results of the antimicrobial activity of the compounds (Table - IV) are active against all the bacteria strains (positive and negative) at varying degrees. Compound 5d, 5e with the electron withdrawing halogen exhibited the highest activity against standard drug Streptomycin is a bacterial and Ketonoazole is a antifungal. Compound 5d exhibited activity against all the bacteria except *S. agalactiae* and also inactive against *S. typhimurium* and *P. mirabilis* at 10mg/ml. Compound 5e showed activity against *K. pneumonia*, *P. aeruginosa*, *S. aureus* and *S. typhimurium* with minimum activity against *E. coli*, *S. agalactiae*. The resistance of the pathogens towards the tested compounds can be attributed to the existence of cell wall in gram positive bacteria which reduces the permeability of the tested compounds, while the activity against them can be attributed to the greater lipophilicity of the compounds.

Table IV: Antimicrobial activity of compounds.

Entry Strains	Antibacterial MIC ($\mu\text{g/mL}$)				Antifungal MIC ($\mu\text{g/mL}$)	
	B. subtilis	S. aureus	P. aeruginosa	E. coli	A. Niger	C. Albicans
5a	05	06	08	05	07	04
5b	17	16	18	17	13	14
5c	18	16	18	14	13	14
5d	21	20	20	21	16	17
5e	20	21	19	19	16	18
5f	10	12	09	10	07	08
Streptomycin	25	25	25	25	-	-
Ketonoazole	-	-	-	-	22	22
DMSO						

4. CONCLUSION

The reaction condition carried out at room temperature for all the newly synthesized compounds. An effective technique for creating a new Schiff base (5a-5f), (E)-5-styryl-1, 3, 4-thiadiazol-2-amine, with P-substituted aromatic aldehyde in the presence P-toluenesulphonic acid at room temperature ethanol as a solvent and the cinnamic acid and with thiosemicarbaide can be converted into the intermediate molecule (E)-5-styryl-1, 3, 4-thiadiazol-2-amine(3) in the presence of acid medium. The yield of the titled compounds

obtained from 83-91%. The compound possesses electron donating group gives maximum yield than that of the compound possesses electron withdrawing group. The rate of reaction developed by using methanesulphonic acid catalyst. All the compounds tested by antimicrobial activity against gram positive, gram-negative and fungal. The compound having electron donating group showed excellent active potential. Otherwise the compounds having halogens which showed better active potential than that of the electron withdrawing group.

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6. Conflict of interest

We declare that we have no conflict of interest

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