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***Research Article***

# Synthesis and characterization of new Schiff base derived from 2-amino-5-(substituted phenyl) thiadiazole, substituted aromatic aldehyde and acetyl acetone

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**Abstract**

All the aimed new Schiff bases are synthesized from 2-amino-5-(substituted phenyl) thiadiazole, substituted aromatic aldehyde and acetyl acetone using silica supported TBAHS as catalyst at reflux temperature and are sufficiently characterized by IR, $^{1}$H NMR and mass spectra.

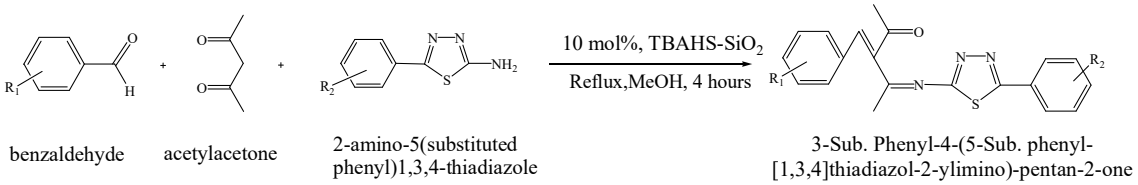
**Keywords**: Thiadiazol; Acetylacetone; Aromatic aldehyde; NMR; Schiff base

## Introduction

Schiff bases play crucial role in biological activity. Schiff bases containing a target molecules works in various types of activities in pharmacology. Mostly, Schiff bases containing azomethine (-C=N-) type of linkage which introduces condensation reaction of carbonyl compound such as aldehydes & ketones with primary aliphatic or aromatic amines. Schiff bases are well known for their antitumor, antifungal, antiviral, antibacterial, and anti-inflammatory activity[1-19]. Today researchers are very much interested to make various types of heterocyclic compounds with help of universal multicomponent route which is more economic, affordable and simple one for the synthesis in one pot manner (without intermediate formation). The activity of Schiff bases were accelerated by forming metal complexes. Metal complexes have been used widely as bactericides, fungicides, insecticide and pesticides[20-27]. Accordingly metal complexes of Schiff bases, containing hetero atoms such as N, S, and O etc. had several biopotencies. Gram negative bacterial infections are very severe than the gram positive bacterial[28-32]. Considering the higher risk for growing antibacterial resistance it is necessary to discover a unique/novel Schiff base with more potent and resistance in pharmacology. Hence, keeping in view of the importance of the Schiff bases we planned to synthesize a library of Schiff bases from 1,3,4-thiadiozole moiety, substituted aldehydes and di-ketones. The derived products were spectroscopically analyzed and also screened by antimicrobial study.

**Present work:**

The synthesis and characterisation of new Schiff base ligands are introduced in this paper. We present a novel approach for synthesising Schiff bases with three components in a single step. Schiff base ligand produced from 2-amino-5-(substituted phenyl) thiadiazole, substituted aromatic aldehyde, and acetyl acetone, which was recently synthesised. The structural characteristics were determined using IR, $^1$H NMR, and Mass. Antimicrobial characteristics of the produced compounds were also tested (**Scheme 1**).




**Scheme 1: TBAHS-catalyzed synthesis of Schiff bases**

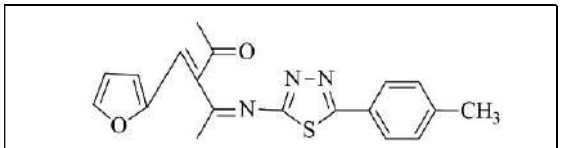
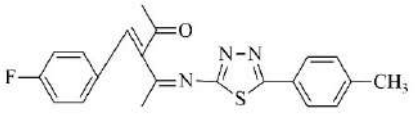
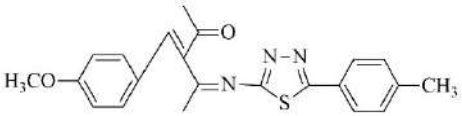
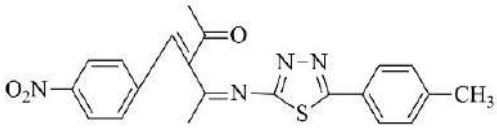
**Experimental**

**Typical procedure of Synthesis of 3-phenyl-4-(5-phenyl-[1,3,4]thiadiazol-2-ylimino)-pentan-2-one**

On a water bath, acetyl acetone (10 mmol), 2-amino-5-(substituted phenyl)thiadiazole (10 mmol), and substituted aromatic aldehyde (10 mmol) were refluxed with 10 mol percent solid supported TBAHS until the reaction was completed. Thin layer chromatography was used to monitor the reaction's completeness (TLC). The catalyst was filtered after the reaction was completed, washed with methanol, and the filtrate concentrated under reduced pressure. Recrystallization with hot methanol purified the crude product. The recovered catalyst was activated for two hours at 180°C and reused four times for complex preparation. For new reactions, the catalyst's activity remains unchanged.

**Result and Discussion**

Commonly, the Schiff bases were obtained by heating under reflux condiation of particular substituted aldyhyde, diketone and substituted thiadiazole in 1:1:1 proporation of molar ratio[33]. The coined Schiff bases purity was determined by TLC using silica gel as adsorbent and solvent system as benzene, acetone (7:3 v/v) ratio. Schiff bases gave a exact melting point showing the purity of ligand. Also molecular weight and elemental analysis of Schiff bases acquired were in good agreement with expected formulas of Schiff bases. The structural, spectroscopic and analytical data were shown as per expected formulae.

| Structure & molecular formula |
|---|
|  $C_{19}H_{17}N_3O_2S$ |
|  $C_{21}H_{18}N_3OSF$ |
|  $C_{22}H_{21}N_3O_2S$ |
|  $C_{21}H_{18}N_4O_3S$ |

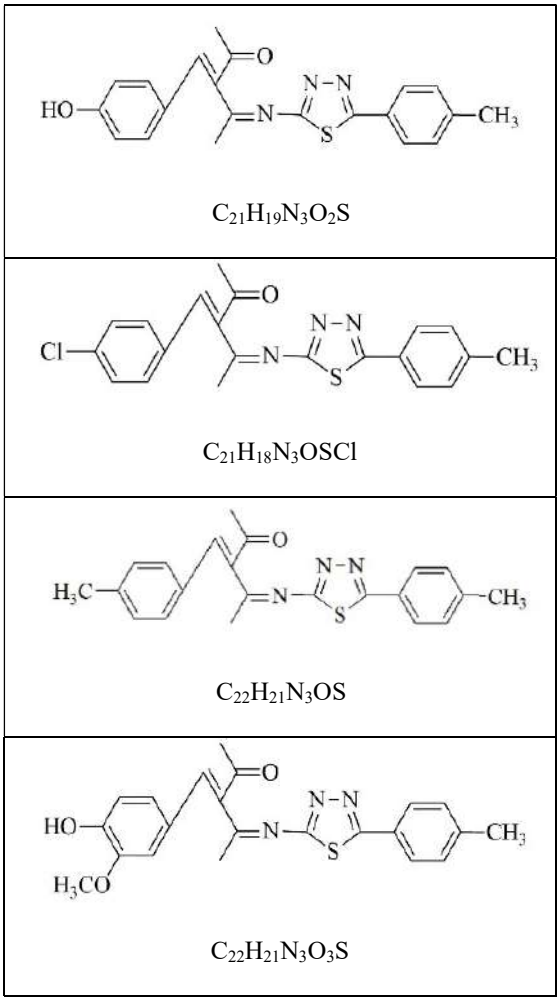



**Fig. 1: Schiff bases derived from 2-amino-5-(substitutedphenyl) thiadiazole, substituted aromatic aldehyde and acetyl acetone**

**Characterization:**

1. *Characterization of 3-(Furan-2-yl methylene)-4-(5-P-tolyl-1, 3, 4 -2-ylimino) Pentane-2-one:*
   Yield: 82%, m. p.107 ($^0$C), IR (KBr) υ cm$^{-1}$ 1708 (C=O), 1633 (C=N), 1020 (N-N), 634 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) $\delta$ 1.80 (S, 3H, N=C-$CH_3$), 2.10 (S, 3H, O=C-$CH_3$), 5.30 (S, 1H, HC=C), 2.41 (S, 3H, Ar-$CH_3$), 6.20-7.80 (M, 8H, Ar-H), Turbo spray MS- m/z 351 $(M+1)^+$, Anal. For $C_{19}H_{17}N_3O_2S$ (%):(C) 65.00, (H) 4.88, (N) 11.97, (S) 9.13, and (O) 9.12.

2. *Characterization of (3E)-3-(4-Fluorobenzylidene)-4-(5-p-tolyl-1,3,4-thiadiazole-2- ylimino) pentane-2-one:*
   Yield: 80%, m. p. 145 ($^0$C), IR (KBr) υ cm$^{-1}$ 1693 (C=O), 1620 (C=N), 1018 (N-N), 632 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) δ 1.91 (S, 3H, N=C-$CH_3$), 2.25 (S, 3H, O=C-$CH_3$), 5.50 (S, 1H, HC=C), 6.30-8.35 (M, 8H, Ar-H), Turbo spray MS- m/z 379 $(M+1)^+$, Anal. For $C_{21}H_{18}N_3OSF$ (%): (C) 66.55,( H) 5.05, (N) 11.09, (S) 8.46, and (O) 8.44.

3. *Characterization of (3E)-3-(4-methoxybenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:*
   Yield: 85%, m. p. 136 ($^0$C), IR (KBr) υ cm$^{-1}$ 1693 (C=O), 1606 (C=N), 1033 (N-N), 634 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) δ 1.89 (S, 3H, N=C-$CH_3$), 2.15 (S, 3H, O=C-$CH_3$), 5.36 (S, 1H, HC=C), 6.89-7.79 (M, 8H, Ar-H),Turbo spray MS- m/z 391 $(M+1)^+$, Anal. For $C_{22}H_{21}N_3O_2S$ (%): (C) 67.40, (H) 5.05, (N) 10.71, (S) 8.17, and (O) 12.23.

4. *Characterization of (3E)-3-(4-notrobenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:*
   Yield: 81.5%, m. p. 138 ($^0$C), IR (KBr) υ cm$^{-1}$ 1726 (C=O), 1662 (C=N), 1012 (N-N), 603 (C-S-C), $^1$H NMR (400 MHz, $CDCl_3$) δ 1.86 (S, 3H, N=C-$CH_3$), 2.19 (S, 3H, O=C-$CH_3$), 5.55 (S, 1H, HC=C), 7.35-8.23 (M, 8H, Ar-H),Turbo spray MS- m/z 406 $(M+1)^+$, Anal. For $C_{21}H_{18}N_4O_3S$ (%): (C) 62.12, H 4.72,( N) 13.80, (S) 7.89, and (O) 15.75.

5. *Characterization of (3E)-3-(4-hydroxybenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:* Yield: 86%, m. p. 132 ($^0$C), IR (KBr) υ cm$^{-1}$ 1704 (C=O), 1635 (C=N), 1062 (N-N), 634 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) δ 1.84 (S, 3H, N=C-$CH_3$), 2.19 (S, 3H, O=C-$CH_3$), 5.55 (S, 1H, HC=C), 7.35-8.23 (M, 8H, Ar-H),Turbo spray MS- m/z 381 $(M+1)^+$, Anal. For $C_{21}H_{19}N_3O_2S$ (%): (C) 66.20, (H) 5.29, (N) 11.02, (S) 8.41, and (O) 12.59.

6. *Characterization of (3E)-3-(4-chlorobenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:*
   Yield: 78.5%, m. p. 142 ($^0$C), IR (KBr) υ cm$^{-1}$ 1707 (C=O), 1660 (C=N), 1012 (N-N), 632 (C-S-C), $^1$H NMR (400 MHz, $CDCl_3$) δ

1.90 (S, 3H, N=C-$CH_3$), 2.22 (S, 3H, O=C-$CH_3$), 5.45 (S, 1H, HC=C), 7.33-8.33 (M, 8H, Ar-H), Turbo spray MS- m/z 395 $(M+1)^+$, Anal. For $C_{21}H_{18}N_3OSCl$ (%): (C) 63.85, H 4.85, (N) 10.63, (S) 8.11, and (O) 8.10.

7. *Characterization of (3E)-3-(4-methylbenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:* Yield: 79%, m. p. 139 ($^0$C), IR (KBr) υ $cm^{-1}$ 1707 (C=O), 1656 (C=N), 1022 (N-N), 634 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) δ 1.82 (S, 3H, N=C-$CH_3$), 2.17 (S, 3H, O=C-$CH_3$), 5.35 (S, 1H, HC=C), 6.90-7.85 (M, 8H, Ar-H),Turbo spray MS- m/z 375 $(M+1)^+$, Anal. For $C_{22}H_{21}N_3OS$ (%): (C) 70.46, (H) 5.91, (N) 11.20, (S) 8.55, and (O) 8.53.

8. *Characterization of (3E)-3-(4-hydroxy-3-methoxybenzylidene)-4-(5-p-tolyl-1,3,4 thiadiazole-2-ylimino)pentane-2-one:* Yield: 87%, m. p. 144 ($^0$C), IR (KBr) υ $cm^{-1}$ 1691 (C=O), 1633 (C=N), 1033 (N-N), 634 (C-S-C), $^1$H NMR (400 M Hz, $CDCl_3$) δ 1.94 (S, 3H, N=C-$CH_3$), 2.22 (S, 3H, O=C-$CH_3$), 5.45 (S, 1H, HC=C), 6.82-7.79 (M, 8H, Ar-H),Turbo spray MS- m/z 407 $(M+1)^+$, Anal. For $C_{22}H_{21}N_3O_3S$ (%): (C) 64.92, (H) 5.45, (N) 10.32, (S) 7.88, and (O) 15.72.

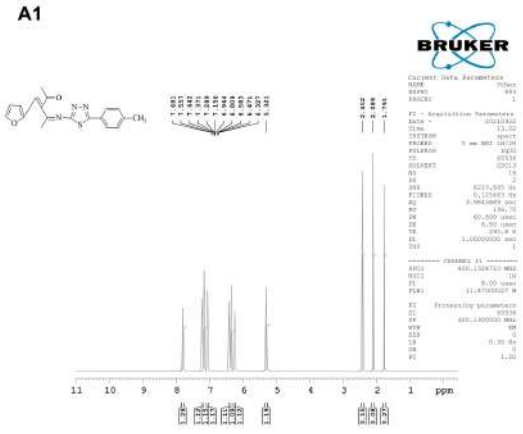



**Fig. 2: $^1$H NMR Spectra of ligand**

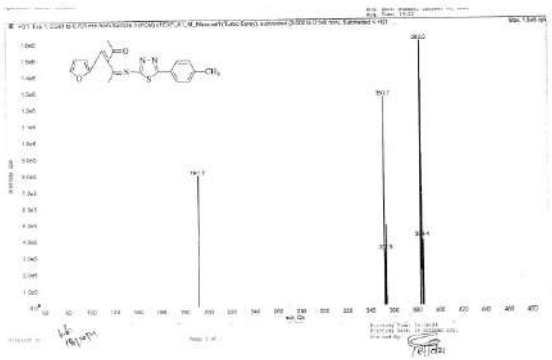


Fig 3 : Mass Spectra of ligand

**Antimicrobial activity:**

The *in-vitro* antibacterial (**Table 1**) and antifungal (**Table 2**) activities of synthesized Schiff bases have been studied by disc diffusion method[34,35]. Four fungal and bacterial strains were tested for antifungal and antibacterial activity at concentrations of 100-200 g/mL in chloroform solvent. Minimum inhibitory concentration method was used to kill *Aspergillus niger*, *Candida albicans*, *Penicillium chrysogenum*, *Rhizopus spp.*, and *Staphylococcus aureus*, *Shigella spp.*, *Escherichia coli*, *Bacillus megaterium*. These bacterial and fungal cultures were cultured at 30°C for one day. Fluconazole and streptomycin, both antifungal and antibacterial, were employed. The diameter of the inhibitory zone was measured to evaluate activity (mm). It is observed that $C_{21}H_{19}N_3O_2S$ and $C_{21}H_{18}N_3OSCl$ ligands are more active against the fungal and bacterial strains *A. Nigar* and *S. aureus* as compare to other fungal and bacterial strains. Other ligands are moderately active against all fungal and bacterial strains[36,37] Antibacterial activities are shown in **Table 2**.

**Conclusion**

In summary, the author has synthesized new Schiff bases from 2-amino-5-(substituted phenyl) thiadiazole, substituted aromatic aldehyde and acetyl acetone using TBAHS catalyst in methanol as solvent at reflux temperature. The synthesized products were evaluated analytically, spectroscopically by elemental analysis FT-IR, $^1$H NMR and mass spectra. All synthesized Schiff bases also screened by antimicrobial activity and found to be moderate to high antifungal and antibacterial activity with respect to standard.

**TABLE 1 : PRELIMINARY IN VITRO ANTIBACTERIAL SCREENING ACTIVITIES OF LIGANDS**

| Compounds | Inhibition Zone (mm) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Antibacterial Activity | | | | | | | |
| | *Staphylococcus aureus* | | *Shigella* spp | | *Escherichia coli* | | *Bacillus megaterium* | |
| | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** |
| **$C_{19}H_{17}N_3O_2S$** | + | + | + | + | + | + | + | + |
| **$C_{21}H_{18}N_3OSF$** | + | + | + | + | + | + | + | + |
| **$C_{22}H_{21}N_3O_2S$** | + | + | + | + | + | + | + | + |
| **$C_{21}H_{18}N_4O_3S$** | + | + | + | + | + | + | + | + |
| **$C_{21}H_{19}N_3O_2S$** | + | ++ | ++ | ++ | ++ | ++ | ++ | ++ |
| **$C_{21}H_{18}N_3OSCl$** | ++ | +++ | ++ | +++ | ++ | ++ | ++ | ++ |
| **$C_{22}H_{21}N_3OS$** | + | ++ | + | ++ | + | + | + | + |
| $C_{22}H_{21}N_3O_3S$ | + | + | + | + | + | + | + | + |
| **+ = 5-10 mm, ++ = 11-20mm, +++ = larger than 20mm and - = no inhibition.** | | | | | | | | |

**TABLE 2 : PRILIMINARY IN VITRO ANTIFUNGAL SCREENING ACTIVITIES OF LIGANDS**

| Compounds | Inhibition Zone (mm) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Antifungal Activity | | | | | | | |
| | *Aspergillus niger* | | *Candida albicans* | | *Penicillium chrysogenum* | | *Rhizopus* spp | |
| | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** | **100 (µg/mL)** | **200 (µg/mL)** |
| **$C_{19}H_{17}N_3O_2S$** | + | + | + | + | + | + | + | + |
| **$C_{21}H_{18}N_3OSF$** | + | + | + | + | + | + | + | + |
| **$C_{22}H_{21}N_3O_2S$** | + | + | + | + | + | + | + | + |
| **$C_{21}H_{18}N_4O_3S$** | + | + | + | + | + | + | + | ++ |
| **$C_{21}H_{19}N_3O_2S$** | ++ | ++ | ++ | ++ | ++ | ++ | ++ | ++ |
| **$C_{21}H_{18}N_3OSCl$** | ++ | ++ | ++ | ++ | ++ | ++ | ++ | +++ |
| **$C_{22}H_{21}N_3OS$** | + | + | + | + | + | + | + | ++ |
| $C_{22}H_{21}N_3O_3S$ | + | + | + | + | + | + | + | + |
| **+ = 5-10 mm, ++ = 11-20mm, +++ = larger than 20mm and - = no inhibition.** | | | | | | | | |

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**Conflict of interest**

The author confirms that the content has no conflict of interest.

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