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# Facile synthesis, characterization, biological studies of novel schiff base ligand and its metal complexes

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An amino molecule and a carbonyl compound are combined to form a Schiff base ligand- 4-[(E)-[(2-hydroxynaphthalen-1-yl)methylidene]amino]-N-(pyrimidin-2-yl)benzene-1-sulfonamide and its metal complexes. This versatile chemical-based has multiple commercial uses. A novel Schiff base ligand and a variety of metal complexes have been created and characterized using elemental analysis, FT-IR, mass, $^{1}H$ and $^{13}C$ NMR analysis. The complexes antioxidant activity was assessed using the the DPPH$^{\bullet}$ free radical scavenging test. The disc diffusion technique was used to test the antibacterial activity of all the compounds. It was discovered that the complexes exhibited greater activity compared to the free ligands.

**Keywords**: *Bacillus amylolique faciens*, *Macrophomina phaseolina,* Metal complexes, Schiff base, *Sclerotium rolfsii*

The potential for designing and developing novel medicinal molecules with outstanding biological activity using the accessible metals is enormous in the context of transition metal complexes[1]. Condensation of molecules produces diverse derivatives like Schiff bases, which have unique biological functions[2-3]. Schiff bases, formed by the reaction of primary amines with aldehydes or ketones, are structurally stable and have various chemical characteristics used in industrial, catalytic and biological sciences[4-9]. Schiff bases and their derivatives have showed promise in medicinal applications, including inhibition of bacterial growth and possible cancer therapy[10-12]. They have analgesic qualities, modulate neurotransmitter pathways and reduce inflammation, providing useful options for pain management[13-15]. Modifying the geometry of the metal complexes with Schiff base ligands makes them more reactive and more physiologically active[16-17]. The biological features of metal complexes are significantly affected by the ionic nature, oxidation state and ligands linked to the metal ions[18-22]. In addition to the availability of the -C=N- group in the Schiff base and various metal ions, the complexes formed from pyrimidine moieties exhibit stupendous biological characteristics, including herbicidal, antibacterial, antifungal, antioxidant and tumor-fighting capabilities[23-28]. The excellent solubility in ordinary solvents and the outstanding stability of the coordination compounds make Schiff bases ideal ligands[29]. An aromatic amine is nucleophile-added to a carbonyl molecule to produce Schiff base[30].

The biological sector has come to recognize the major relevance of cobalt and its complexes, which are crucial components of coenzyme $B_{12}$[31-33]. These complexes have been studied for their antioxidant, antiproliferate, antibacterial and antiviral properties[34-39]. There has been a lot of focus on developing efficient techniques for synthesizing Schiff bases because of their wide range of uses in chemistry[40-44]. We intend to construct Schiff base transition metal complexes from 4-amino-N-(pyrimidin-2-yl)benzene-1-sulfonamide, 2-hydroxynaphthalene-1-carbaldehyde and their different metal(II) and (IV) salts, based on the aforementioned facts and their applications.

## Materials and Methods

All of the reagents and compounds were acquired from TLC and utilized in our research without any additional purification. The synthesis and recrystallization processes were carried out using solvents of AR grade. Analytical grade chemicals were used for every aspect. The components carbon,

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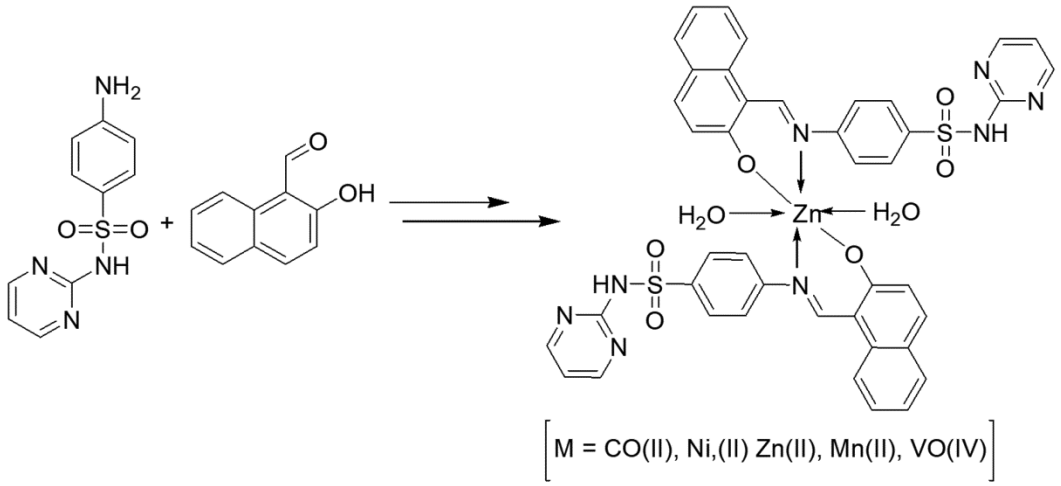



Graphical abstract

hydrogen and nitrogen concentrations were measured using an elemental analyzer called a Carlo Erba1106. The KBr pellets were subjected to infrared spectra recording from 4000-400 $cm^{-1}$ using a Nicolet FT-VERTEX 70 spectrophotometer. A Mercury plus DMSO 400 MHz NMR spectrometer, using TMS as the internal standard, was used to generate the $^{1}H$ NMR and $^{13}C$ NMR spectra.

**Synthesis of Schiff base ligand**

The morphological flexibility, unique structure, and molecular environment sensitivity of a Schiff base composed of nitrogen and oxygen are all significant. In this study, we detail the synthesis of metal chelates of sulfonamide Schiff base ligands. At temperatures ranging from 60 to $800^{0}C$ the 4-amino-N-(pyrimidin-2-yl)benzene-1-sulfonamide reacts with 2-hydroxynaphthalene-1-carbaldehyde to form the equivalent ligand 4-[(E)-[(2-hydroxynaphthalen-1-yl)methylidene]amino]. N-(pyrimidin-2-yl)benzene-1-sulfonamide has been explained.

Yeild: 85%, $T_{m.p}$: 176.21-177.32°C, IR (KBr)υ: 3307 (OH), 3458 (NH), 1342 (CN) $cm^{-1}$. $^{1}H$ NMR spectrum, δ, ppm: 5.28 (s, 1H, OH), 6.73 (m, 3H, Ar-H), 7.43 (m, 5H, Ar-H), 7.70 (s, 1H, NH), 8.09 (m, 5H, Ar-H), 8.39 (s, 1H, CH); $^{13}$CNMR spectrum, δ C,ppm: 116.40, 118.20, 121.06, 121.51, 125.40, 127.71, 128.23, 129.92, 130.34, 134.58, 135.39, 154.54, 155.78, 158.51, 163.44: Mass spectrum: *m*/*z* 404.09, Anal. Cacld for $C_{21}H_{16}N_4O_3S$; C 62.36; H 3.99; N 13.85; Found: C 62.38; H 4.02; N 13.89.

**Synthesis of metal complexes**

In order to synthesize complexes with transition metals like CO(II), Ni(II), Zn(II), Mn(II) and VO(IV) a 1:2 metal-to-ligand ratio is essential. The ethanolic solution of the ligand (100 mmol) that had been magnetically refluxed was combined with a 50 mmol salt solution in ethanol. The product precipitated after the reaction mixture was continually refluxed for 6 to 8 hours. The filtered product was further refined by washing it with hot ethanol.

**CO(II)complex**

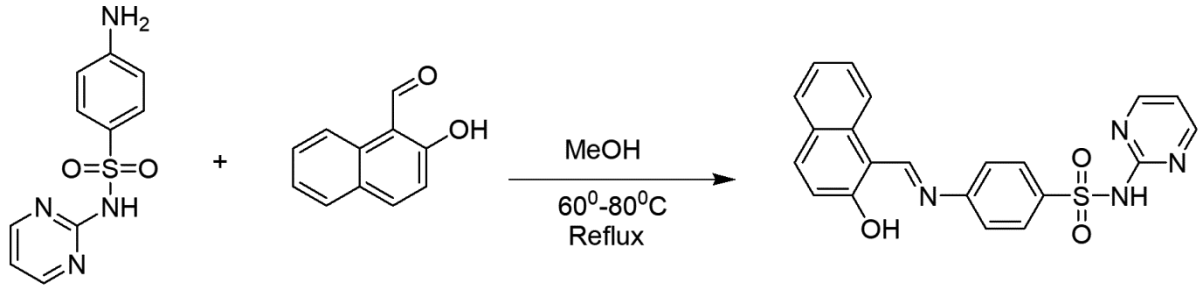
Yeild: 68%, $T_{m.p}$: >300°C, IR (KBr)υ: 3350 (OH), 3400 (NH), 1320 (CN) $cm^{-1}$. Mass spectrum: *m*/*z* 901.01, Anal. Cacld for $C_{42}H_{34}CoN_8O_8S_2$; C 55.94; H 3.80; N 12.42; Found: C 55.95; H 3.82; N 12.45.

**Ni(II)complex**

Yeild: 65%, $T_{m.p}$: >300°C, IR (KBr)υ: 3450 (OH), 3350 (NH), 1340 (CN) $cm^{-1}$. Mass spectrum: *m*/*z* 901.06, Anal. Cacld for $C_{42}H_{34}N_8NiO_8S_2$; C 55.95; H 3.80; N 12.43; Found: C 55.97; H 3.81; N 12.45.

**Zn(II) complex**

Yeild: 68%, $T_{m.p}$: >300°C, IR (KBr)υ: 3200 (OH), 3420 (NH), 1385 (CN) $cm^{-1}$. $^{1}H$ NMR spectrum, δ, ppm: 6.73 (m, 3H, Ar-H), 7.21 (m, 4H, Ar-H), 7.52 (m, 5H, Ar-H), 7.63 (m, 5H, Ar-H), 7.70 (s, 2H, NH), 7.92 (m, 5H, Ar-H), 8.09 (m, 4H, Ar-H), 8.38 (s, 4H, Ar-H), 8.79 (s, 2H, CH); $^{13}C$ NMR spectrum, δ C, ppm: 108.60, 110.32, 116.40, 118.20, 118.40, 118.42, 121.06, 121.51, 122.52, 122.63, 123.92, 125.40, 126.53, 126.62, 126.86, 127.71, 128.02, 128.23, 128.69, 129.13, 129.92, 130.34, 132.42, 132.53, 134.58, 135.39, 138.26, 138.35, 153.72, 154.54, 155.78, 156.42, 157.92, 158.51, 160.10, 160.32, 163.44, 169.32, 169.52: Mass spectrum: *m*/*z* 908.15, Anal. Cacld for $C_{42}H_{34}N_8O_8S_2Zn$; C 55.54; H 3.77; N 12.34; Found: C 55.56; H 3.79; N 12.36.




Scheme 1 — Synthesis of sulfonamide Schiff base ligand and their corresponding metal complexes

**Mn(II) complex**

Yeild: 69%, $T_{m.p}$: >300°C, IR (KBr)υ: 3320 (OH), 3250 (NH), 1350 (CN) $cm^{-1}$. Mass spectrum: *m/z* 897.82, Anal. Cacld for $C_{42}H_{34}MnN_8O_8S_2$; C 56.19; H 3.82; N 12.48; Found: C 56.21; H 3.84; N 12.50.

**VO(IV) complex**

Yeild: 70%, $T_{m.p}$: >300°C, IR (KBr)υ: 3360 (OH), 3400 (NH), 1310 (CN) $cm^{-1}$. Mass spectrum: *m/z* 909.08, Anal. Cacld for $C_{42}H_{34}N_8VO_9S_2$; C 55.44; H 3.77; N 12.32; Found: C 55.58; H 3.79; N 12.34.

## Results and Discussion

A nitrogen and oxygen Schiff base is morphologically versatile, has a distinctive structure, and is susceptible to molecular environments. The development of sulfonamide Schiff base ligands and thereafter their metal chelates has been reported in this research work. The 4-amino-N-(pyrimidin-2-yl)benzene-1-sulfonamide[45] is condensing with 2-hydroxynaphthalene-1-carbaldehyde at 60 to 80°C reflex with MeOH will give corresponding ligand 4-[(E)-[(2-hydroxynaphthalen-1-yl)methylidene]amino]-N-(pyrimidin-2-yl)benzene-1-sulfonamide (Scheme 1).

The synthesis of complexes using transition metals, such as CO(II), Ni(II), Zn(II), Mn(II) and VO(IV) are required a 1:2 ratio of metal to ligand. The ethanolic solution of the ligand (100 mmol) that had been magnetically refluxed was mixed with a 50 mmol solution of salt in ethanol. After refluxing the resulting reaction mixture continuously for 6 to 8 h, the product precipitated. The filtered product was further purified by being washed with the heated ethanol (Fig. 1).

**Antioxidant activity**

The synthesised metal complexes (1−5) were tested for their antioxidant activity using the stable 2, 2-Diphenyl-2-picryl-hydrazyl (DPPH) free radical in accordance with the Blois technique with a few modifications. The UV-Vis spectrophotometer was

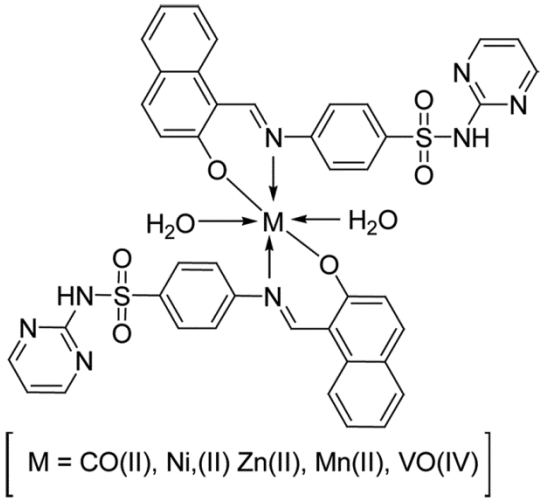



Fig. 1 —Types of Metal complexes

used to examine the metal complexes antioxidant activity in a 0.3 μM concentrated DPPH (DMSO) solution. Because it contains an odd electron, DPPH exhibits a wide and robust absorption band at 517 nm. All of the metal complexes (1–5) and standard solutions (20–100 μM) of DPPH (0.3 μM) were produced using about 1 mL of solutions. These solutions were given a good shake after addition and then left to incubate at room temperature for 40 minutes under darkness. Following that, measurements of absorbance at 517 nm were taken. The $IC_{50}$ values (50% Inhibitory Concentration) were used to clarify the antioxidant activity of metal complexes. All drugs $IC_{50}$ values were calculated using line regression techniques.

**Antimicrobial activity**

This study involved conducting an *in vitro* antimicrobial test on two Gram negative bacteria, *Pseudomonas aeruginosa* and *Escherichia coli*, as well as two Gram positive bacteria, *Staphylococcus aureus* and *Bacillus amylolique faciens*. The synthesised Schiff base ligand along with their metal complexes (1−5) were the subjects of the test. Two

Table 1 — Antioxidant activity results of Schiff base ligand and its metal complexes

| S. No. | Compound | DPPH/Inhibition % | $IC_{50}$ (μL) |
|---|---|---|---|
| 1 | Ligand | 48.1 ± 0.09 | - |
| 2 | CO(II) | 45.0 ± 0.03 | - |
| 3 | Ni(II) | 43.4 ± 0.02 | - |
| 4 | Zn(II) | 55.2 ± 0.02 | 117.4 |
| 5 | Mn(II) | 55.3 ± 0.06 | 111.6 |
| 6 | VO(IV) | 57.0 ± 0.07 | 113.2 |
| 7 | Ascarbic acid | 57.2 ± 0.08 | 115.0 |

--Not inhibited

Table 2 — Antimicrobial activity results of Schiff base ligand and its metal complexes

| S. No. | Bacterium (mm) | | | | Fungi (mm) | |
|---|---|---|---|---|---|---|
| Compound | Gram-positive bacteria | | Gram-negative bacteria | | | |
| | *Bacillus Amyloliquefaciens* | *Staphylococcus aureus* | *Escherichia coli* | *Pseudomonas aeruginosa* | *Sclerotium rolfsii* | *Macrophomina phaseolina* |
| Ligand | 09 | 10 | 09 | 10 | 12 | 13 |
| CO(II) | 12 | 12 | 13 | 10 | 13 | 12 |
| Ni(II) | 15 | 16 | 19 | 15 | 18 | 15 |
| Zn(II) | 16 | 18 | 16 | 16 | 19 | 17 |
| Mn(II) | 15 | 14 | 12 | 10 | 16 | 15 |
| VO(IV) | 13 | 12 | 10 | 09 | 12 | 14 |
| Streptomycin | 12 | 11 | 16 | 11 | - | - |
| Mancozeb | - | - | - | - | 19 | 18 |

fungal strains, *Macrophomina phaseolina* and *Sclerotium rolfsii*, were studied using the disc diffusion technique.

Metal complexes (1-5) and Schiff bases ligand have their inhibition zone values shown in (Table 1). When tested against several bacterial and fungal strains, the Schiff base ligands showed modest activity. However, when combined with metal complexes, they exhibited increased activity compared to free Schiff base ligands. In contrast to free Schiff base ligands, their metal complexes exhibited greater activity against bacterial and fungal strains, while the former were only modestly effective. After analysing the data, it is clear that Ni(II) and Zn(II) complexes have the greatest ability to combat the specified bacterial and fungal species. Also included in the study is the data reported in (Table 2), which pertains to the minimum inhibition concentration (MIC) approach.

## Conclusion

The synthesis, characterization and Biological studies of Schiff base ligands and their complexes are investigated in this work. The synthetic approach in this investigation resulted in molecules having a molar ratio of 1:2 (L:M). The azomethine nitrogen and phenolic oxygen nuclei coordinate the metal ion, resulting in a stable heterocyclic ring by deprotonation. The chelation theory suggests that this synthetic substance has antimicrobial and antioxidant functions. Furthermore, the findings showed that the complexes under investigation had a greater effect on Gram-positive bacteria and Gram-negative bacteria. The antibacterial activity of the compounds may be related to the structure of the bacterial cell wall.

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## Conflict of interest

All authors declare no conflicts of interest.

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