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SYNTHESIS, CHARACTERIZATION, SPECTRAL AND ANTIFUNGAL STUDIES OF Ni(II) AND Zn(II) COMPLEXES DERIVED FROM 2-AMINOPYRIMIDINE-2-THIOPHENECARBOXALDIMINE

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ABSTRACT
Schiff bases are an important class of compounds characterized by the presence of an imine (–C=N–) functional group, which imparts remarkable coordination ability towards metal ions. In the present work, Schiff bases were synthesized through the condensation of aldehydes with primary amines, followed by the synthesis of their corresponding Ni(II) and Zn(II) metal complexes. The synthesized compounds were characterized using standard physicochemical and spectroscopic techniques to confirm their structures. The antifungal activities of both Schiff bases and their Ni(II) and Zn(II) complexes were evaluated against selected fungal strains. The results revealed that the metal complexes exhibited enhanced antifungal activities compared to the free Schiff bases, indicating that complexation plays a crucial role in improving biological efficacy. This study underscores the potential of Ni(II) and Zn(II) Schiff base metal complexes as promising candidates for the development of novel antifungal agents.

KEYWORDS: Schiff Base, Metal Complexes, Synthesis, Antifungal Activities.

INTRODUCTION
Schiff bases with aryl substituents are more stable and easier to synthesize, while those with alkyl groups or aliphatic aldehydes are less stable and prone to polymerization.[3,4] Aromatic aldehyde Schiff bases, stabilized by conjugation, are more robust, as aldehydes react faster than ketones due to lower steric hindrance and higher electrophilicity. Schiff bases remain attractive because of their simple synthesis, availability, and versatile electronic features, with applications across organic,[5] inorganic,[6,7] coordination,[8-10] bioinorganic[11,12] and environmental chemistry,[13-16] as well as in medicine, catalysis, metallurgy, sensing, and diagnostics.

A major milestone in coordination chemistry, Schiff bases readily form stable complexes with transition metals due to their strong chelating ability. The azomethine nitrogen, with its sp^2 lone pair, plays a key role in coordination, while additional groups like –OH and –SH enable bi- or multidentate binding. Schiff bases with heterocyclic rings, offering multiple donor atoms are particularly significant.[17-19]

Among pyrimidine derivatives, 2-aminopyrimidine is notable for its ambident nucleophilicity and hydrogen bonding. Its Schiff bases exhibit antifungal, antimicrobial,[20] antitubercular,[21] and antitumor[22] activities, and the C-2 amino group allows condensation with aldehydes/ketones to give ligands for coordination complexes.[23-25] These complexes are recognized for redox activity, DNA-binding, and cytotoxicity.[26-27] Likewise, 2-amino-4,6-dihydroxypyrimidine introduces extra hydroxyl groups that enhance solubility, delocalization and biological interactions.[28-29] Schiff bases of this scaffold show antibacterial, antifungal and anticancer activity via DNA binding and enzyme inhibition.[30-34]

EXPERIMENTAL

All the chemicals used were of AR/GR grade. Pure sample of 2-aminopyrimidine (AP), molecular formula $C_4H_5N_3$, molecular weight 95.10 g/mol, melting point~122-126^oC was obtained from SRL Chemicals Ltd. Metal salts such as $ZnCl_2$, $NiCl_2.6H_2O$ were of Merck. Solvents used were ethanol, acetone, DMF and dimethylsulfoxide (DMSO).

Synthesis of Schiff base

The compound was synthesized from 2-thiophenecarboxaldehyde and 2-aminopyrimidine by adding 1000 ml of 2-thiophenecarboxaldehyde ethanolic solution (1.22g; 0.01mol) to same volume of ethanol solution/same solvent of 2-aminopyrimidine (0.8144g;0.01mol), the mixture was refluxed for 2 hour and kept overnight at room temperature. The resulting solution was evaporated to 20% of its original solution, and the product was collected by filtration, washed several times with ethanol, recrystallized from hot ethanol and then dried. The melting point of off white crystals was found to be 175°C.

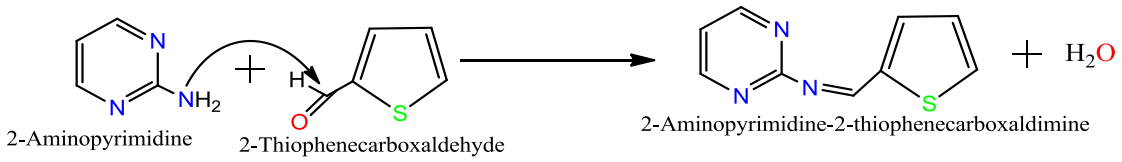


Fig. 1: Synthesized Schiff Base.

Synthesis of Complexes

The solid complexes were prepared by mixing, ethanolic solutions of the ligand (HL) (0.18 g and 0.01mol) with ethanolic solution of chlorides of Ni(II) (0.059 g and 0.005 mol), Zn(II) (0.049 g and 0.005 mol) separately. The resulting solutions were checked for pH and pH was adjusted by adding few drops of N/10 NaOH solution. The solutions were refluxed for 4 hrs and the refluxed solutions were kept for some days, solid crystalline complexes appeared in the solution which were filtered off, washed thoroughly with same solvent and finally with acetone, vacuum dried and weighed. Melting points of the complexes were recorded.

Analysis and Instrumentation

Elemental analysis was carried out on Vario MICRO V2.20 Elementar Analyse Systeme GmbH, from IIIM, Jammu. Metal contents were determined gravimetrically.[35] The infrared spectra were recorded on FT-IR Spectrophotometer Model RZX (Perkin Elmer) using KBr pellets from SAIF, Panjab University, Chandigarh. Mass were recorded at CIL, Panjab University, Chandigarh by (LC-MS Spectrometer Model Q-TOF Micro Waters). Thermogravimetric measurement were carried out at Department of Chemistry, IICT Hyderabad in nitrogen atmosphere (0.00 l/min and 11/min).

RESULTS AND DISCUSSION

The Schiff base ligand (L) was synthesized by the condensation of the amino group of 2-aminopyrimidine with 2-thiophenecarboxaldehyde. This ligand was then reacted with metal ions to yield Schiff base metal complexes. Both the ligand and the metal(II) complexes were obtained in good yields from ethanol and were isolated in pure form. The ligand appeared off-white in color, while the nickel and zinc complexes were light brown and dark brown, respectively.

Elemental analyses of the complexes were consistent with a 1:2 metal-to-ligand stoichiometry, leading to the general formula $[M(L_2)]$ (M = Ni(II), Zn(II)). The complexes are air-stable solids at room temperature and do not decompose over time. They are non-hygroscopic, insoluble in water and most common organic solvents, but soluble in polar solvents such as DMF and DMSO. The molar conductance values of the complexes, measured in 10^{-3} M DMF, fall in the range of[35-49] Ω^{-1} cm^2 mol^{-1}, confirming their non-electrolytic nature.[36] Magnetic studies revealed that the nickel complex is paramagnetic, while the zinc complex is diamagnetic. The detailed physico-chemical characterization, analytical data, and molar conductance values are summarized in Table 1 and Table 2.

Table 1: Physico-Chemical Characteristics of Ligand APT and its Complexes.

S. No	Ligand/Complexes	Colour	% Yield	M.P ^oC	Molar Conductance Ω^{-1} cm^2 mol^{-1}	Magnetic Moment B.M
1	APT	Yellow	73	175	-	-
2	$[Ni(APT)_2]$	Greyish brown	69	260 (Decomposed)	15.3	3.80
3	$[Zn(APT)_2]$	Beige brown	63	292-300 (Decomposed)	16.6	0.0

Table 2: Analytical Data of APT and its Complexes.

S. No	Molecular Formula (Molecular Weight)	Elemental Analysis (%) Found (Calculated)				
		C	H	N	S	Metal
1	$C_9H_7N_3S$ (189.24)	57.12 (57.02)	3.72 (3.69)	22.20 (22.17)	16.94 (16.89)	- -
2	$C_{18}H_{14}N_6S_2Ni$ (437.17)	49.45 (49.24)	3.22 (3.19)	19.22 (19.15)	14.66 (14.59)	13.42 (13.38)
3	$C_{18}H_{14}N_6S_2Zn$ (443.86)	48.70 (48.6)	3.17 (3.15)	18.93 (18.9)	14.44 (14.4)	14.72 (14.71)

Magnetic Measurements

The Ni(II) complex showed a value of 3.80 B.M., which is slightly lower than the spin only value of 3.20 B.M. for high spin octahedral Ni(II) complexes.[37] Zn(II) complex showed magnetic moment values of 0.0 B.M., indicating diamagnetic nature of Zn(II) ion.[38-39]

FTIR Spectra

The IR spectra of the complexes indicate that the Schiff base acts as a tridentate ligand, coordinating through thiophenesulfur, azomethine nitrogen and pyrimidine nitrogen. In the free ligand (APT), the $\upsilon_{(-HC=N)}$ band appears at 1622 cm^{-1}, which shifts to 1617 cm^{-1} in $[Ni(APT)_2]$ and 1619 cm^{-1} in $[Zn(APT)_2]$, confirming the involvement of azomethine nitrogen in coordination.[40-42] Further M-N bonding evidence comes from the bands at 436 cm^{-1} and 490 cm^{-1} $\upsilon_{(M-N)}$, for Ni(II) and Zn(II) complexes, confirming metal–ligand coordination.[43] Additionally, the bands at 1063 cm^{-1} for $[Ni(APT)_2]$ and 1053 cm^{-1} for $[Zn(APT)_2]$ are attributed to $\upsilon_{(M-S)}$, supporting the involvement of sulfur in coordination.[44]

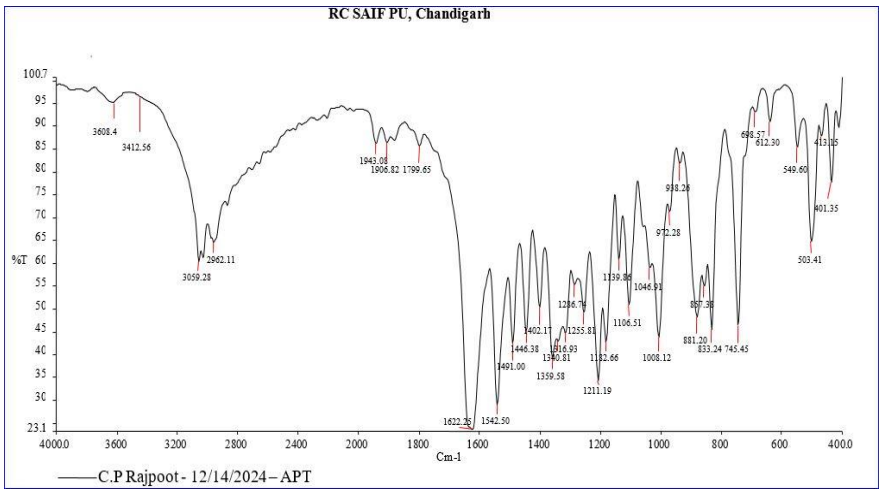


Fig. 2: IR Spectra of Schiff Base (APT)

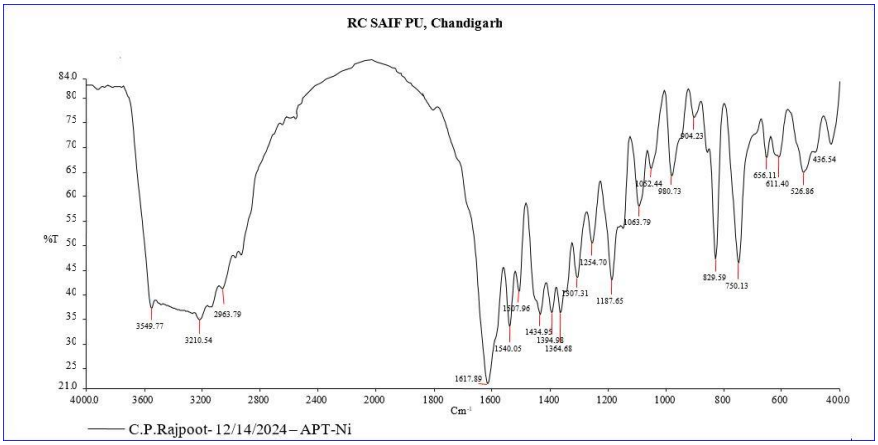


Fig. 3: IR Spectra of Schiff Base Derived Complex (APT-Ni)

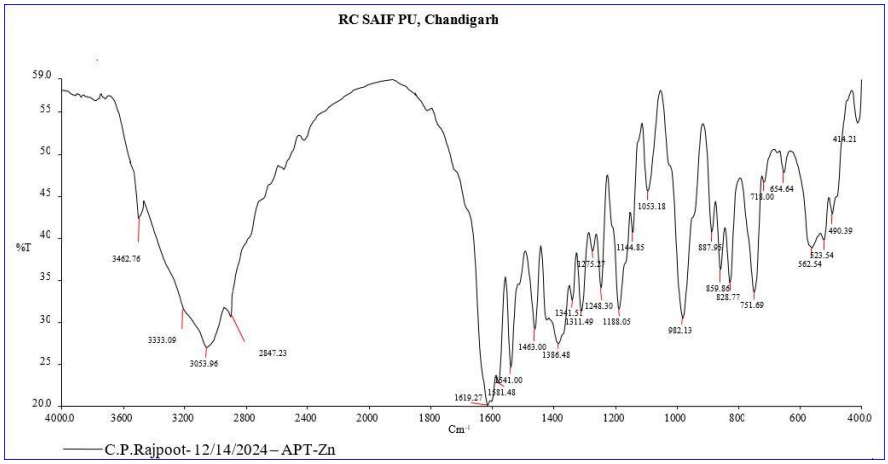


Fig. 4: IR Spectra of Schiff Base Derived Complex (APT-Zn).

Table 3: FTIR data for APT and its metal complexes.

S. No	Groups	APT	$[Ni(APT)_2]$	$[Zn(APT)_2]$
1	$\upsilon_{(HC=N)}$	1622 s	1617 s	1619 s
2	$\upsilon_{(M-N)}$	-	436 m	490 m
3	$\upsilon_{(S-M)}$	-	1063 s	1053 s

s = strong, m = medium , b = broad, w= weak

Mass Spectra

The mass spectrum of the ligand (Schiff base) $C_9H_7N_3S$ shows a molecular ion peak (m^+) at m/z 189.24 due to $(L)^+$ that corresponds to the molecular weight of the Schiff base. Fragment ion peaks values at m/z 163.2, 137.18, 64.07, and 38.05 is due to detachment of ions like $(C_2H_2)^+$, $(CN)^+$,$(C_2H_2NS)^+$,$(CN)^+$ from the synthesized ligand of APT.

The mass spectrum of $[Zn(C_{18}H_{14}N_6S_2)]$ shows a molecular ion peak at m/z 443.86, confirming the proposed formula. Fragmentation gives peaks at m/z 254.62 (loss of $C_9H_7N_3S$), 228.06 (loss of –CN), 189.24 (loss of $C_9H_7N_3S$–Zn), 162.19 (loss of C_2H_3), 136.18 (loss of –CN), and 26.04 (loss of C_4H_2NS, indicating pyrimidine ring cleavage). The peak intensities reflect the abundance and stability of fragments, consistent with earlier reports.[45–47]

X-Ray diffraction studies

X-ray powder diffraction pattern is a set of lines or peaks, each of the different intensity and position (d-spacing or Bragg angle, θ) on either a strip of photographic film or on a length of chart paper. For a particular substance, the line positions are essentially fixed and are characteristic of that substance.[48] Each crystalline substance has its own characteristic powder diffraction pattern which may be used for its identification. Standard patterns are given in the powder diffraction file known as the JCDPDS file or formally as the ASTM file.[49]

X-ray diffraction of the Schiff bases and metal complexes derived from these Schiff bases are entirely different (Fig 2 and Table 4). All the reflections in the complexes are new ones and the patterns are also new ones and fairly strong which suggests that there is a complete conversion of reactants into products.[50-53]

Table 4: X-ray diffraction.

2theta(θ)	FWHM
16.45965	0.80955
19.29859	0.2787
22.94538	0.20434
23.65983	0.25602
27.16143	0.48861
28.96637	0.16589
32.1889	0.77888
34.39413	0.80543
36.53252	0.46939
41.32371	0.31189
43.50218	0.36174
46.99367	0.30287
53.3866	0.37692

59.1120	0.02662
62.34533	64.6763
67.97627	0.36724
76.31306	0.49166

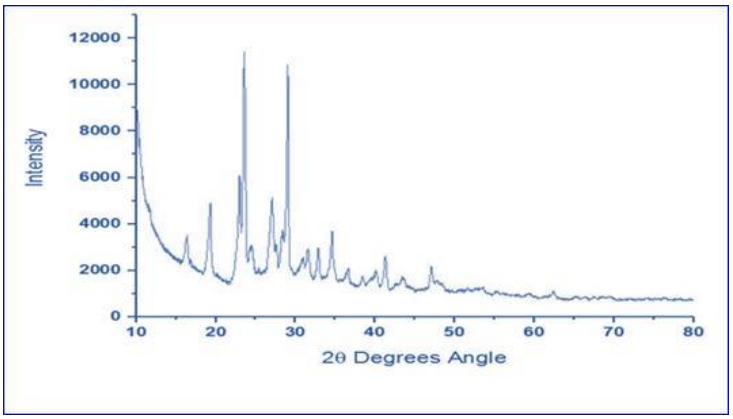


Fig. 5: X-ray diffraction graph.

Antifungal Activity

The antifungal activity of 2-aminopyrimidine-2-thiophenecarboxaldimine and its metal complexes was evaluated *in vitro* by the agar well diffusion method against fungal species such as *R. nigricum* and *C. albicans*. The antifungal screening results (Table 5) reveal that the free ligand (APT) exhibits only mild activity against *R. nigricum* (1.38 mm) and *C. albicans* (1.20 mm). However, metal complexation significantly enhances the activity. The Ni(II) complex shows the highest inhibition values (3.21 mm and 3.45 mm), which are comparable to the standard drug griseofulvin (3.46 mm and 3.90 mm). The Zn(II) complex displays moderate inhibition (1.80 mm and 1.75 mm), higher than the free ligand but lower than the Ni(II) complex. The improved activity of the Ni(II) complex may be explained by chelation, which increases lipophilicity and facilitates penetration through fungal cell membranes. Thus, [APT–Ni] demonstrates promising antifungal potential, approaching that of the reference drug.

Table 5: Zone of inhibition for Schiff base APT and its metal complexes (mm).

Compound	*R.nigricum*	*C.albicans*
APT	1.38	1.2
APT-Ni	3.21	3.45
APT-Zn	1.8	1.75
GRISEOFULVIN	3.46	3.9

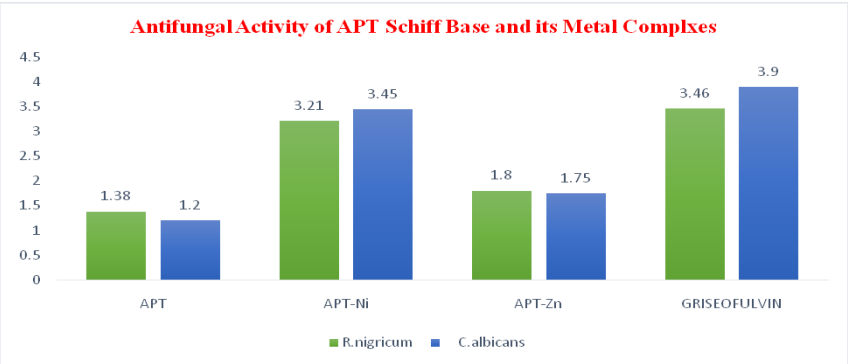
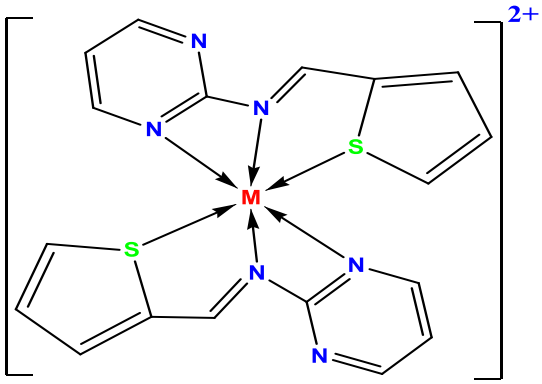


Fig. 6: Antifunagal activity of APT and derived complexes.

CONCLUSION

Based on stoichiometry and analytical data, it is concluded that the ligand is neutral, tridentate coordinating through the "N," "N," and "O" of the azomethine group, pyrimidine ring, and phenolic group, respectively. All the complexes possesses 2:1 (L:M) stoichiometry based on analytical and spectral data and octahedral structures have been proposed for the complexes. The ligand and the complexes showed very good activity against all bacteria.



M= Ni(II) and Zn(II)

Fig. 7: Proposed Structure of the complexes

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