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Original Article

Coordination Scaffolds of Schiff Base Derived from Imatinib Amine: Synthesis, Spectroscopic and *in vitro* Biological Screening

Prashant Gajera[1,*], Milan Vadodaria[2], Rahul Parmar[3]

[1]Department of Chemistry, Atmiya University, Rajkot, Gujarat, INDIA.
[2]Department of Chemistry, Chemical Research Laboratory, Shri M. and N. Virani Science College, Rajkot, Gujarat, INDIA.
[3]Department of Chemistry, Faculty of Science, Monark University, Ahmedabad, Gujarat, INDIA.

ABSTRACT

Aim: Imatinib amine based Schiff base heterochelates: Synthesis, Spectroscopic and *in vitro* biological screening. **Materials and Methods:** We acquired 2-Hydroxy-5-bromo benzaldehyde and 2-Hydroxy-5-nitro benzaldehyde from Sigma Ltd. (India). Purchased from Almon Industries in Ahmedabad, Gujarat, India, imatinib amine was utilised unpurified. **Results:** Elements C, H and N were elementally analysed using a Perkin-Elmer model 2400 elemental analyzer; ^{1}H NMR spectra were obtained with an Advance 400 Bruker FT-NMR apparatus in DMSO-d_6 solvent; FT-IR spectra were obtained as KBr pallets with a Nicolet-400D spectrophotometer; and the complex's FAB-mass spectrum was measured with a JEOL SX-102/DA-6000 mass spectrometer. **Conclusion:** As part of our present research, we are developing new Schiff's base ligands based on imitinib amine and their heterochelates with transition metals. All of the ligands and heterochelates that were synthesised were validated and characterised using ^{1}H NMR, IR and mass spectrometry. Every synthesised chemical was tested for its ability to fight germs. The heterochelates have potent antibacterial action against Gram-positive (*Staphylococcus epidermidis, Bacillus subtilis*) and Gram-negative (*Escherichia coli, Salmonella enterica*) pathogens in contrast to their corresponding ligands. The heterochelates show significant effectiveness against one or more bacteria, so providing a new class of bactericidal drugs based on transition metals for investigation in the future.

Keywords: Antimicrobial studies, Imatinib amine, Schiff base, Spectroscopic studies, Transition metal-based coordination compounds.

Correspondence:
Dr. Prashant Gajera
Assistant Professor, Department of Chemistry, Atmiya University, Rajkot-360005, Gujarat, INDIA.
Email: ps9601477204@gmail.com

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INTRODUCTION

The chemical compounds known as Schiff Bases (SBs) are defined by the presence of an imine or azomethine group (>C=N-). These compounds have extensive use as colours and tints, catalysts, polymer stabilisers,[1] luminescence chemo devices[2] and organic synthesis intermediates.[3] Additionally, SBs may be utilised in various metal-electrolyte systems as corrosion inhibitors since they absorb and create an anti-corrosion surface layer via their electron-rich centres, such as the imine moiety. Actually, due of its π-acceptor characteristics, this moiety may provide a strong bonding with metallic ions.[4-6] Additionally, a number of research examined SBs' tribological properties and their use as biolubricant additives.[7-10]

The availability, electrical characteristics and ease of synthesis of transition metals have made coordination chemistry of Schiff bases a popular topic of study in recent years. Due to its many applications-including thermal research,[11] catalysis,[12] toxicity,[13] optically active materials,[14] agriculture,[15] anti-bacterial activity,[16] anti-fungal action,[17] anti-cancer action,[18] anti-viral movement,[19] and DNA binding-Schiff base coordination chemistry has recently become more and more well-known.[20] The C=N (Imine) moiety of these molecules determines their biological action.

Motivated by the implications of transition metal-based research, we present the coordination scaffolds of Schiff base produced from Imatinib amine: synthesis, spectroscopy and *in vitro* biological screening. These days, coordination chemistry of Schiff bases is a prominent subject of study due to the availability, electrical properties and simplicity of synthesis of transition metals. This has piqued our interest in the science of drug-based Schiff base chemistry.

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MATERIALS AND METHODS

Materials

Since every component utilised was of analytical quality, no further purification was necessary. Sigma Ltd., provided the ortho-Vanillin and Salicylaldehyde (India). Without purification, Imatinib amine was utilised after being purchased from Almon Industries in Ahmedabad, Gujarat, India.

Finding techniques

A Perkin-Elmer model 2400 elemental analyser was used for elemental analysis (C, H and N). ^{1}H NMR spectra were obtained using the latest 400 Bruker FT-NMR device in deuterated Di Methyl Sulfoxide flush. FT-IR bands were recorded using a Nicolet-400D spectrophotometer as KBr pallets. The FAB-mass spectrum of the complex was measured by a JEOL SX-102/DA-6000 mass spectrometer.

Common process for synthesis of Ligands (L_1-L_2)

A 1:1 molar methanolic solution of Imatinib amine (0.001 mol) was whirled in binary necked curved end flasks at reflux temperature for few min. The aforementioned solution was then mixed with a methanolic solution of salicylic aldehyde and its derivative (0.001 mol) drop-wise with continuous stirring at 60°C and refluxed for 4 hr. Accomplishment of the reaction was confirmed using TLC. The product was immediately permitted to stand at room temperature after the reaction. In order to achieve dense material, the solid product was finally crystallised using methanol and then rinsed with diethyl ether.

RESULTS

L_1

Yellow powder, Yield 81%; M.P. 204°C; Exact mass calcd. For $C_{23}H_{19}N_5O$; 380.50; found 381; IR (KBr, cm^{-1}): (3040) N-H, (3237) O-H, (1591)ˀC=N; ^{1}H NMR (400 MHz, DMSO) δ: 2.29 (3H, s, -CH_3); 6.79-7.73 (7H, c, Ar-H); 8.41-9.28 (6H, c, Ar-H of Pyridine and Pyrimidine ring of Imatinib amine), Elemental analysis originate (%) C, 72.48; H, 5.07; N, 18.40 intended for $C_{23}H_{19}N_5O$: C, 72.42; H, 5.02; N, 18.36.

L_2

Orange powder, Yield 67%; M.P. 210°C; Exact mass calcd. For $C_{24}H_{21}N_5O_2$; 410.20; found 411; IR (KBr, cm^{-1}): (2985) N-H, (3351) O-H, (1583)ˀC=N; ^{1}H NMR (400 MHz, DMSO) δ: 2.29 (3H, s, -CH_3); 3.81 (3H, s, -OCH_3); 6.86-7.73 (6H, c, Ar-H); 8.41-9.27 (6H, c, Ar-H of Pyridine and Pyrimidine ring of Imatinib amine), Elemental analysis originate (%) C, 70.10; H, 5.19; N, 17.05 intended for $C_{24}H_{21}N_5O_2$: C, 70.06; H, 5.14; N, 17.02.

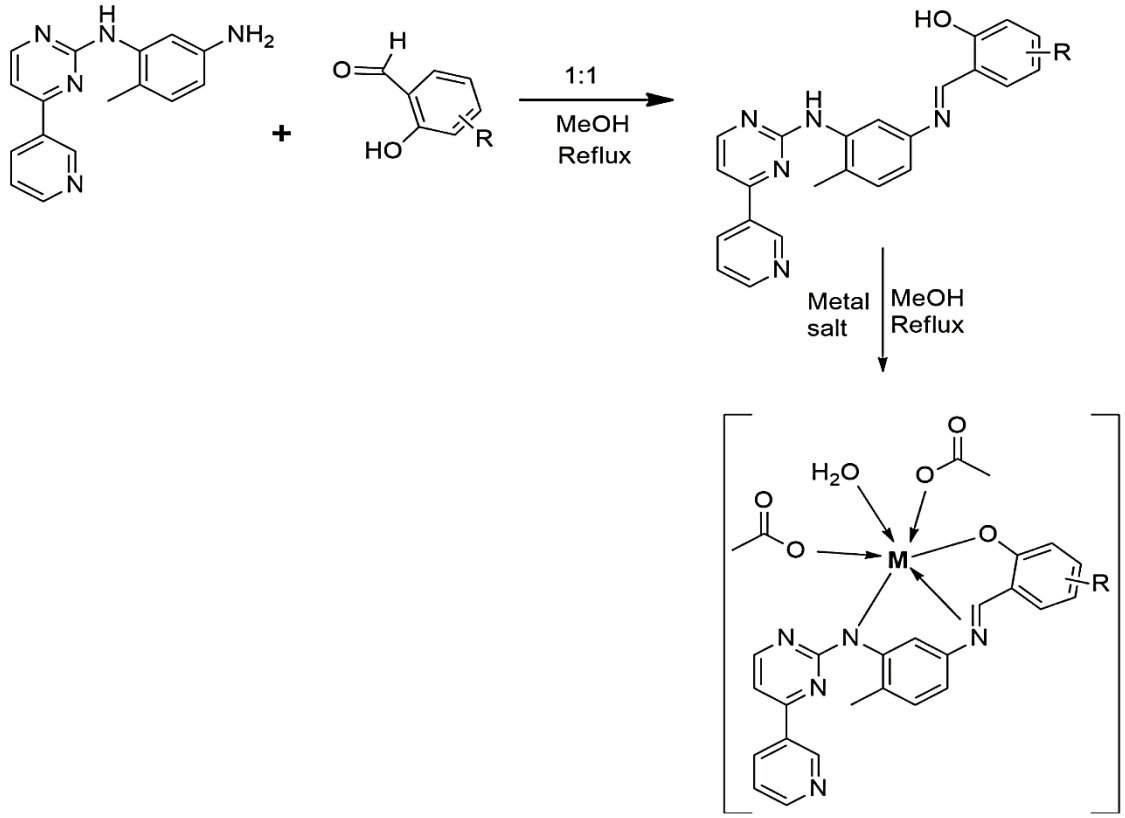

Figure 1: Reaction Scheme for the synthesis of ligands and it's heterochelates. [Where R=H for L_1 and R=OCH_3 for L_2].

Common process for the synthesis of metal coordination scaffolds (ML_1-ML_2)

Each complex has been isolated and synthesised using a generic technique. A warm methanolic solution of various transition metal (II) acetate salt (0.001 mol) was added gradually drop by drop continuous stirring in 1:1 molar ratio to the corresponding ligand solution (0.001 mol). After 4 hr of heating to 70°C, the combination was promptly allowed to calm to room temperature. The highly pigmented creation was soaked in methanol and water and then allowed to dry in desiccators. Methods for the preparation and proposed structure of coordination compound is shown in Figure 1.

In vitro biological screening

The biological screening *in vitro* was the standard turbidimetric approach was used to analyse the *in vitro* biological screening. The samples were checked using a combination of Gramme positive bacteria (*Staphylococcus epidermidis*, MTCC 3615), Gramme negative bacteria (*Escherichia coli*, MTCC 739) and Gramme positive bacteria (*Bacillus subtilis*, MTCC 441). The bacterial growth media used was nutrient broth. Traditionally, streptomycin and chloramphenicol were employed as antibiotics to combat microbes. The biological action of several combinations melted in DMSO at a concentration of 1000 µg/mL was evaluated using actively developed culture bacteria that had been cultured for 18 hr. At 37°C, the bacterial culture was incubated. After a 24 hr period, a turbidimetric study of the suppression of bacterial culture development was conducted. Activity of combinations was measured by change of growth based on measured optical density.

Table 1: Analytical and physical data of heterochelates.

Compounds Chemical Formula	Formula Weight	Colour	(%Yield)	Analysis (%) Found (Cal)			
				C	H	N	M
$[Mn(L_1)(CH_3COO)_2(H_2O)]C_{27}H_{25}N_5O_6Mn$	570	Brown	83	56.88(56.85)	4.44(4.42)	12.31(12.28)	9.66(9.63)
$[Ni(L_1)(CH_3COO)_2(H_2O)]C_{27}H_{25}N_5O_6Ni$	574	Brown	81	56.51(56.48)	4.41(4.39)	12.24(12.20)	10.25(10.22)
$[Cu(L_1)(CH_3COO)_2(H_2O)]C_{27}H_{25}N_5O_6Cu$	579	Green	87	56.05(56.00)	4.38(4.35)	12.12(12.09)	11.00(10.97)
$[Zn(L_1)(CH_3COO)_2(H_2O)]C_{27}H_{25}N_5O_6Zn$	581	Yellow	85	55.85(55.83)	4.36(4.34)	12.09(12.06)	11.29(11.26)
$[Mn(L_2)(CH_3COO)_2(H_2O)]C_{28}H_{27}N_5O_7Mn$	600	Brown	84	56.05(56.01)	4.57(4.53)	11.69(11.66)	9.18(9.15)
$[Ni(L_2)(CH_3COO)_2(H_2O)]C_{28}H_{27}N_5O_7Ni$	604	Black	69	55.69(55.66)	4.53(4.50)	11.63(11.59)	9.75(9.71)
$[Cu(L_2)(CH_3COO)_2(H_2O)]C_{28}H_{27}N_5O_7Cu$	609	Brown	72	55.25(55.21)	4.50(4.47)	11.53(11.50)	10.46(10.43)
$[Zn(L_2)(CH_3COO)_2(H_2O)]C_{28}H_{27}N_5O_7Zn$	611	Black	79	55.09(55.05)	4.49(4.45)	11.48(11.46)	10.73(10.70)

Table 2: IR data of ligands and its metal heterochelates.

Compounds	v(C=N)	v(N-H)	v(O-H)	v(C=O) Acetate
L_1	1591	3040	3237	-
$[Mn(L_1)(CH_3COO)_2(H_2O)]$	1567	-	-	1709
$[Ni(L_1)(CH_3COO)_2(H_2O)]$	1580	-	-	1698
$[Cu(L_1)(CH_3COO)_2(H_2O)]$	1587	-	-	1702
$[Zn(L_1)(CH_3COO)_2(H_2O)]$	1581	-	-	1704
L_2	1583	2985	3351	-
$[Mn(L_2)(CH_3COO)_2(H_2O)]$	1551	-	-	1708
$[Ni(L_2)(CH_3COO)_2(H_2O)]$	1556	-	-	1701
$[Cu(L_2)(CH_3COO)_2(H_2O)]$	1556	-	-	1712
$[Zn(L_2)(CH_3COO)_2(H_2O)]$	1557	-	-	1706

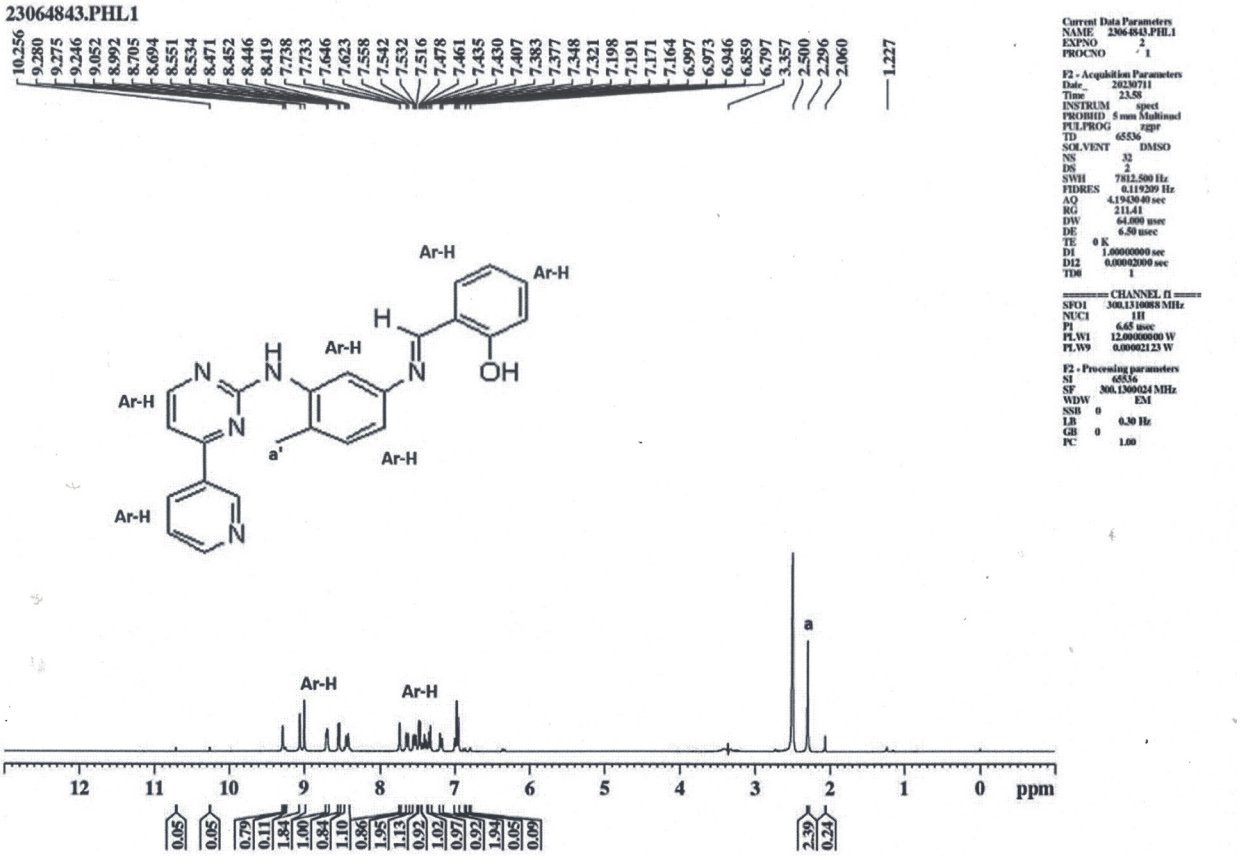

Figure 2: ^{1}H NMR spectrum of Ligand L_1.

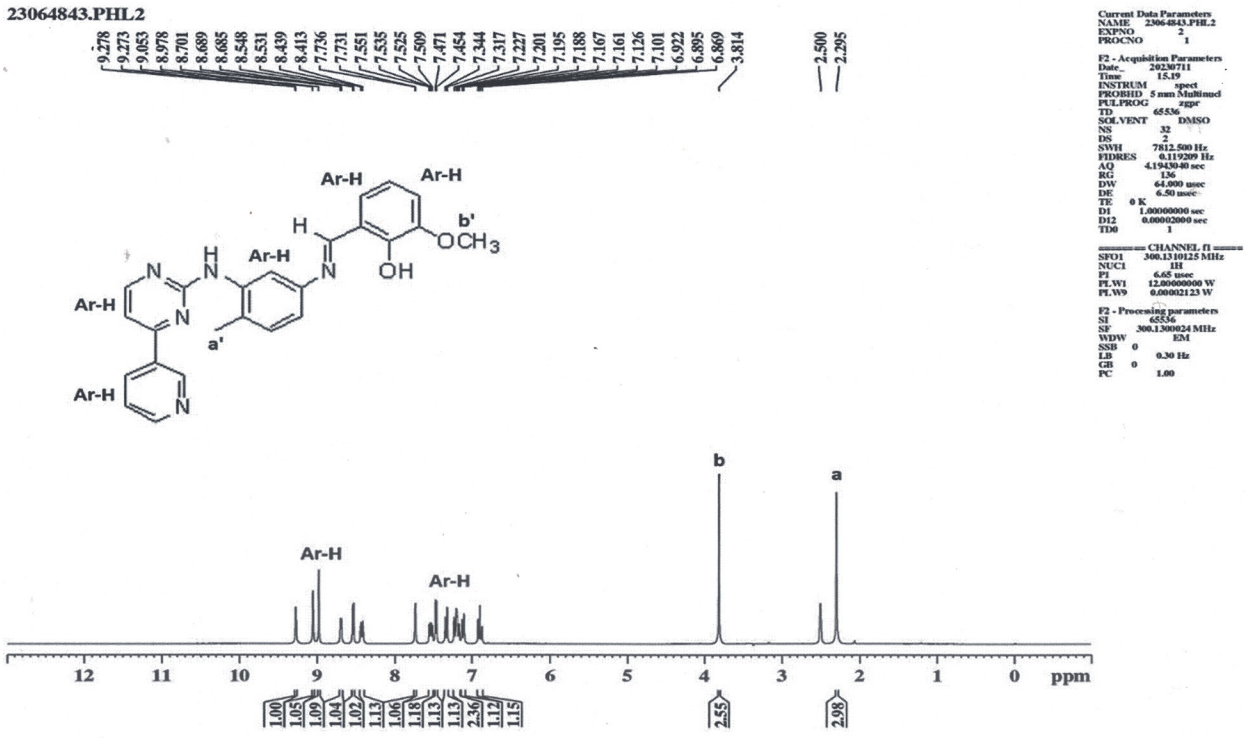

Figure 3: ^{1}H NMR spectrum of Ligand L_2.

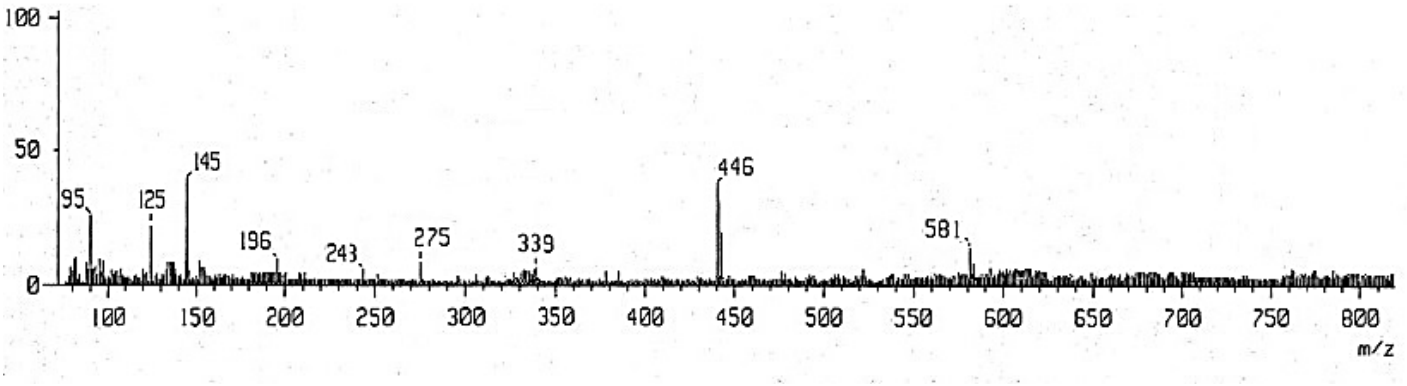

Figure 4: FAB Mass spectrum of complex $[Zn(L_1)(CH_3COO)_2(H_2O)]$.

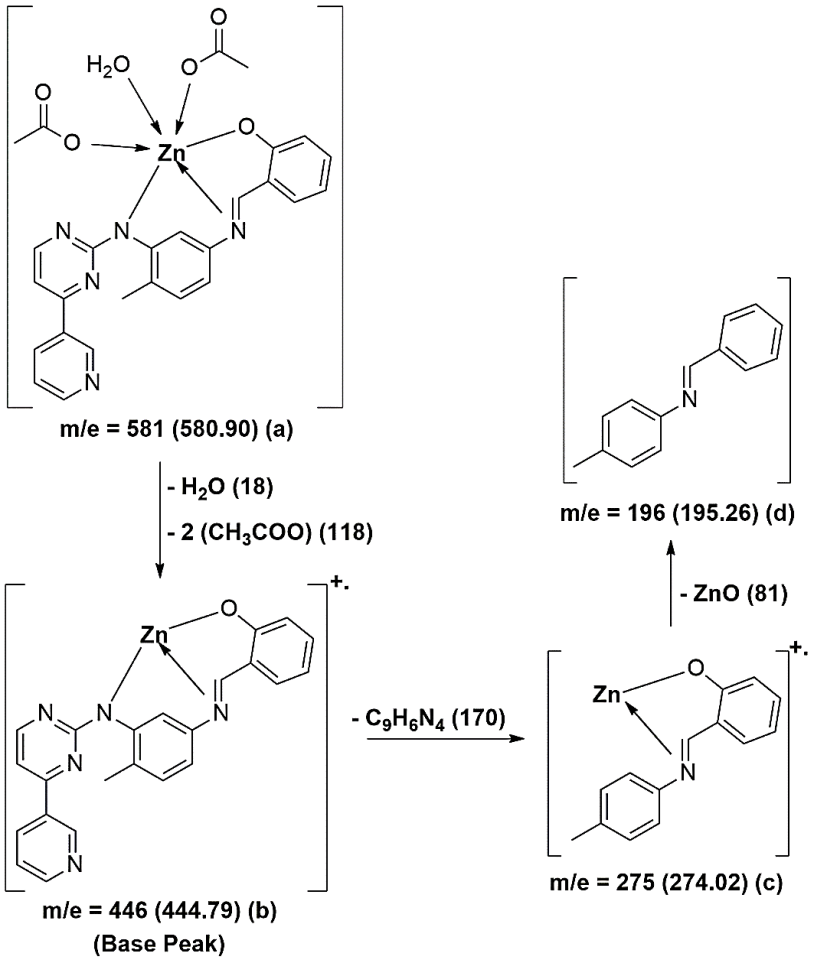

Figure 5: Proposed mass fragmentation pattern for complex $[Zn(L_1)(CH_3COO)_2(H_2O)]$.

MIC Study

Each chemical's Minimum Inhibitory Concentration (MIC) was considered by means of the microtitre broth dilution method with both Gramme +ve and Gramme -ve bacteria. Among the prescribed drugs were streptomycin, chloramphenicol and nystatin. At doses of 1000 µg/mL, 500 µg/mL, 250 µg/mL and 100 µg/mL, each tester was diluted with DMSO. The negative control is made up of chemicals in DMSO solution, whereas the positive control is made up of microorganisms that are not taking medicine. Actively developing standard microbial cultures were cultivated in nutrient broth medium. The biological strain was grown at 37°C. After a day, absorbance for bacterial cultures was restrained at 600 nm by a microplate reader.

DISCUSSION

All of the synthesised Schiff base ligands and complexes had their structures identified by elements analysis, ^{1}H NMR, FAB-Mass spectra and infrared spectra. The experimental part contains the

Table 3: MIC results of the ligands and its heterochelates.

Sl. No.	Compounds	Gramme +Ve (mM)		Gramme -Ve (mM)	
		Staphylococcus epidermidis	*Bacillus subtilis*	*Escherichia coli*	*Salmonella enterica*
Ref. Drug	Streptomycin			100	100
	Chloramphenicol	100	100		
	Nystatin				
1	L_1	1000	500	500	1000
2	$[Mn(L_1)(CH_3COO)_2(H_2O)]$	1000	1000	100	100
3	$[Ni(L_1)(CH_3COO)_2(H_2O)]$	1000	250	1000	1000
4	$[Cu(L_1)(CH_3COO)_2(H_2O)]$	1000	1000	100	1000
5	$[Zn(L_1)(CH_3COO)_2(H_2O)]$	1000	500	1000	100
6	L_2	1000	1000	250	1000
7	$[Mn(L_2)(CH_3COO)_2(H_2O)]$	1000	500	1000	100
8	$[Ni(L_2)(CH_3COO)_2(H_2O)]$	1000	500	1000	1000
9	$[Cu(L_2)(CH_3COO)_2(H_2O)]$	1000	1000	500	1000
10	$[Zn(L_2)(CH_3COO)_2(H_2O)]$	1000	500	1000	500

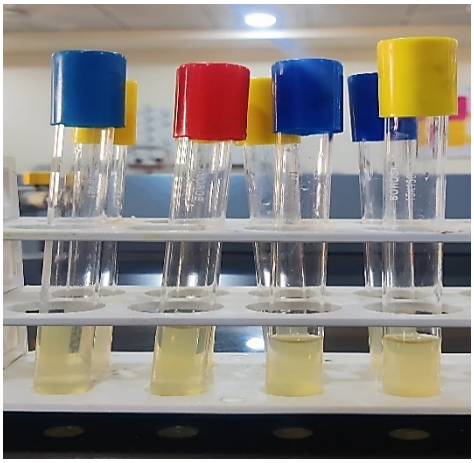

Figure 6: MIC results (µg/mL) of Ligands and its complexes.

^{1}H NMR data for Schiff's base ligands. The complexes' analytical and physical characteristics are displayed in Table 1. The complexes were very slightly soluble in methanol, but completely soluble in DMSO and DMF. For very some time, none of the complexes moved during the airborne phase.

^{1}H NMR

Figures 2 and 3 display the ^{1}H NMR spectra of ligands L_1 through L_2, respectively. The methyl group protons (-CH_3) are responsible for a singlet signal in the 2.29-2.31 δppm range observed in the ^{1}H NMR ranges of ligands L_1-L_2. The presence of the -OCH_3 group is indicated by an additional singlet peak at 3.81 δppm for three protons in the ^{1}H NMR ranges of ligand L_2. The range of aromatic protons in all ligands is 6.79 to 9.28 δppm. It may be difficult to distinguish the signal for the -NH proton in the aromatic region due to mixing. The number of protons precisely corresponds to the molecule's chemical formula.

IR (Infrared Spectra)

Table 2 presents the IR facts for SB Ligands (L_1-L_2) and their transition metal coordination compounds. To determine the ligands' metal binding modes, the Infrared (IR) data of the complexes and the ligands were compared. The acyclic azomethine group's υ(C=N) is clearly seen as a robust band in the 1583-1601 cm^{-1} range for the Schiff base Ligands (L_1-L_2). This band arises between 1537 and 1587 cm^{-1} in the complexes; its reported lower energy shift suggests azomethine nitrogen coordination.[21-23] Acetate binding in complex structure is shown by the prominent υ(C=O) acetate band in complex spectra (1696-1713 cm^{-1}), which is barely discernible in ligand spectra. However, in the 2985-3046 cm^{-1} range, the anilinic -NH band emerged and the amidic-NH_2 band vanished, indicating the development of coordination compound.[24,25]

FAB Mass

$[Zn(L_1)(CH_3COO)_2(H_2O)]$ molecular ion peak and the confirmed mass spectra (Figure 4) were approved the molecular formula for the suggested coordination compound. Figure 5 shows the proposed fragmentation pattern. The complex's molecular ion peak is shown by the first peak at *m/z*=581. The possible breakdown mechanism for the chemical under study is shown in Figure 5. The loss of a water molecule and two CH_3COO molecules from species (a) causes the primary fragmentation of the complex; this leads to species (b), whose base peak of maximum intensity is located at *m/z*=446. Subsequent breakdown results in species (c), where the residual part of the molecule $C_9H_6N_4$ is lost. ZnO loss may be the cause of the decline of species (c) into a stable species

(d). For every suggested degradation process, the calculated molecular weights matched the expected values perfectly.[26,27]

Biological Screening

Development inhibition was detected to preliminary confirms the antimicrobial movement of selected combinations by comparison with standard antimicrobial agents against 02 Gramme +ve and 02 Gramme -ve germs as shown in Table 3. Out of total 10 combinations 03 showed considerable antimicrobial activity as shown in Figure 6. Compounds L_1, $[Cu(L_1)(CH_3COO)_2(H_2O)]$ and $[Mn(L_1)(CH_3COO)_2(H_2O)]$ exhibited highest antibacterial activity against bacteria and particularly against Gramme negative bacteria (100 µg/mL), therefore deliberated as most effective broad-spectrum combination at 500 µg/mL concentration. Compound $[Cu(L_1)(CH_3COO)_2(H_2O)]$ and $[Mn(L_1)(CH_3COO)_2(H_2O)]$ were found effective for *B. subtilis* inhibition however there all over activity against tested bacterial cultures were found poor.

The lowest medication concentration that prevents the development of microorganisms is known as the minimum inhibitory concentration, or MIC. Absorbance value of individual drug at altered dilution was equated with control. The lowest dilutions of antibiotic, which indicate microbial development equated to the positive control were reflected as an actual inhibitory concentrations. Compounds L_1, $[Cu(L_1)(CH_3COO)_2(H_2O)]$ and $[Mn(L_1)(CH_3COO)_2(H_2O)]$ considerable antibacterial potential against Gramme negative cultures (100 µg/mL). Compound L_8 showed the highest inhibition of Gramme-positive *B. subtilis* (250 µg/mL). Other tested compounds are found weak for their antimicrobial potential.

CONCLUSION

We are currently developing novel Schiff's base ligands based on Imatinib amine and their transition metal coordination compounds. ^{1}H NMR, IR and mass spectrometry was used to verify and characterise the synthesised ligands and coordination compounds. We checked each synthesised chemical for the existence of antibacterial properties. The coordination compounds show strong antibacterial action against Gramme +ve (*S. epidermidis, B. subtilis*) and Gramme -ve (*E. coli, S. enterica*) pathogens when compared to their respective ligands. The compounds' promising efficacy against one or more microbes opens the door to future research on a prospective category of antibacterial drugs based on transition metals.

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ABBREVIATIONS

UV-vis: Ultraviolet-visible; **FT-IR:** Fourier Transform Infrared; **NMR:** Nuclear Magnetic Resonance; **MIC:** Minimum Inhibitory Concentration; **TLC:** Thin Layer Chromatography; **FAB:** Fast Atom Bombardment; **DMSO:** Dimethyl Sulfoxide; **MTCC:** Microbial Type Culture Collection; **Zn:** Zinc; **Mn:** Manganese; **Ni:** Nickel; **Cu:** Copper; **IR:** Infrared; **CO_2:** Carbon Dioxide; **TD-DFT:** Time-Dependent Density Functional Theory; **DMOl3:** Density Functional Theory Molecular Orbital Package; **CT-DNA:** Calf Thymus Deoxyribonucleic Acid; **AIE:** Aggregation-Induced Emission; **HCl:** Hydrochloric Acid; **Mg-Zn-Y-Nd:** Magnesium-Zinc-Yttrium-Neodymium; **ACS:** American Chemical Society; **RSC:** Royal Society of Chemistry; **H_2O:** Water; **CH_3COO:** Acetate; ***S. epidermidis*:** *Staphylococcus epidermidis;* ***B. subtilis*:** *Bacillus subtilis*; ***E. coli*:** *Escherichia coli;* ***S. enteric*:** *Salmonella enterica.*

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

SUMMARY

The study focuses on the synthesis, characterization and biological evaluation of imatinib amine-based Schiff base heterochelates. The Schiff base ligands were synthesized through a condensation reaction involving imatinib amine and various aldehydes. These ligands were further complexed with transition metal ions (e.g., Mn(II), Ni(II), Cu(II), Zn(II)) to form heterochelates.

The complexes were characterized using various spectroscopic techniques, including UV-vis, FT-IR, NMR and elemental analysis, confirming their structures and coordination geometry of the synthesized compounds.

The *in vitro* biological activities of compounds were screened against selected microbial strains. The results demonstrated promising biological potential, with certain metal complexes exhibiting enhanced activity compared to the free Schiff base ligands. These findings highlight the potential applications of such complexes in medicinal chemistry.

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