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Comparative Study of Antimicrobial Activity of Novel Schiff Base Ligand and its Metal Complexes Derived from 2-Amino Benzothiazole and Isophthalaldehyde

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ABSTRACT- Microwave assisted, Solvent Free, eco-friendly synthesis of novel Schiff base ligand was synthesize by using 2-Amino Benzothiazole and Isophthalaldehyde and its transition metal complexes were prepared from Ni(II) chloride and Mn(II), Fe(III), Cd(II), Cu(II), Zn(II), Co(II), Ag(I) nitrate. The metal complexes characterized by UV-Visible spectroscopy, IR spectroscopy, and thermo gravimetric analysis. The metal complexes exhibit coordination number 6, so metal complexes exhibit octahedral geometry. The complexes are coloured and stable at room temperature. The Schiff Base ligand and its metal complexes were screened for their antibacterial activity against the gram -positive bacteria Staphylococcus aureus, Gram-negative bacteria Salmonella Typhi and fungi Aspergillus Niger After the antimicrobial activity results it indicates that the metal complexes are better antimicrobial agents as compared to its novel Schiff base ligand.
Keywords: Antimicrobial Activity, Microwave method, Staphylococcus aureus, Salmonella Typhi, Aspergillus Niger

1. Introduction

Microwave assisted solvent-free synthesis approach in today's condition is proved to be most helpful tool for chemical synthesis. This helps to protect the environment from various chemical hazards [1, 2]. Microwave assisted synthesis was introduce in 1955 [3]. Microwave assisted synthesis is easy, solvent free, eco-friendly, less time consuming and giving more yield [4, 5]. It is more convenient for separation and purification [6]. The Schiff base ligand and its metal complexes were prepared by using microwave assisted synthesis. The Schiff bases are formed by action of carbonyl compound with primary amine. The Schiff base contain azomethine (C=N) functional group [7-9]. The Schiff base ligand and its transition metal Complexes have many applications in biological and analytical field [10, 11]. Biological activities include anticancer, and plant growth inhibitor [12-14], bactericidal [15-16], .analgesic [17-18], antiviral [19-20], anti-hypersensitive activity [21], antibiotics [22], insecticidal efficiency [23], and anti-tuberculosis, anticonvulsant drug activity [24-25], anti-inflammatory [26-27].They also used as dyes, catalyst, intermediate and stabilizers[28]. Due to easy and simple condition of work, less reaction time and more yields, this approach is used [29].

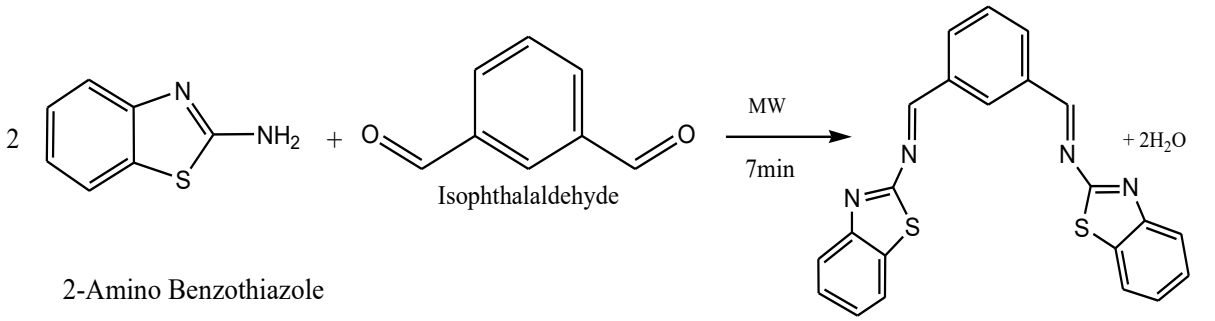
2. Material and Method

All chemicals used are of analytical grade reagents. All the chemicals were purchased from Sigma Aldrich, Loba chem and Merck chemicals. Melting point was recorded on electro-thermal digital apparatus. The ^{1}HNMR were recorded On a Brukers (400MHz, 100MHz) NMR spectrometer. Tetramethylsilane (TMS) was used as the internal standard and the chemical shift

is measured in ppm. LC-MS is used for find out the molecular weight of novel Schiff base ligand. In the wavelength range 200-800 nm. Electronic absorption spectra were recorded using UV-visible Spectrophotometer. The Shimadzu Dr.8031 instrument was used for Infrared spectral studies. The Perkin Elmer thermal analyzer was used for thermo gravimetric analysis of the Complexes. TGA was carried out in dynamic nitrogen atmosphere.

2.1 Synthesis of novel Schiff base ligand

The novel Schiff base ligand was synthesized by the reaction of 2-Amino Benzothiazole and Isophthalaldehyde. The reaction was carried out in scientific microwave oven for 7 minutes at 750 W. The color change was observed. The melting point is recorded after recrystallization. Recrystallization was done with dry ether. The product was confirmed by repeating the synthesis process. The yield obtained was 90%. The melting point recorded was 205℃ . The purity of product was observed by using TLC. The solvent mixture used for TLC was n-hexane and ethyl acetate (7:3). The TLC spot was visualized under UV light.



2-Amino Benzothiazole

Isophthalaldehyde

(8*E*)-*N*-(3-((*E*)-(benzo[*d*]thiazol-2-ylimino)methyl)benzylidene)benzo[*d*]thiazol-2-amine

2.2 Synthesis of metal complexes

The metal complexes were synthesized by mixing the metal nitrates and chloride with the required amount of ligand in 1:2 metal ligand ratios. The reaction mixture was irradiated at 750 W in scientific microwave oven. The irradiated product was washed with ether and filter through whatman filter paper. The final product was recrystallized from absolute ethanol. The metal salts used were hydrated nickel nitrate, ferric nitrate, cadmium nitrate, copper nitrate, zinc nitrate, cobalt nitrate, anhydrous silver nitrate and manganese chloride.

3. Result and Discussion

It was observed that by using microwave assisted synthesis high yield was obtained in very short time compared to conventional method. By rotation of reaction platform tray the unity of the reaction mixture was increased. This is two step reactions. In first step Schiff base ligand was prepared by irradiating 2-Amino Benzothiazole with Isophthalaldehyde. In second step mixture of novel Schiff base ligand and metal salt was irradiated to get the desired metal complex. The entire metal complexes are colored, solid and stable at room temperature. They possess sharp melting point. The complexes are insoluble in common solvent but soluble in DMF and DMSO. The product was confirmed by repeating the synthesis process [30].

3.1 Elemental analysis.

Table 1. Elemental Analysis Data

Compound	Molecular weight	% of C found (Cal.)	% of H found (Cal.)	% of N found (Cal.)	% of S found (Cal.)
$C_{22}H_{14}N_4S_2$	384	67.90 (68.73)	03.80 (3.67)	15.10 (14.57)	13.20 (13.03)

3.2 Physical properties.

Table 2. Physical Properties Data

Sr. no.	Ligand/ Complex	Color	Melting point (°C)	% Yield
1	$(C_{22}H_{14}N_4S_2)$	Yellow	205	90
2	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Ni$	Green	195	90
3	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Mn$	Yellowish brown	240	87
4	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Fe$	Dark brown	293	88
5	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Cd$	Faint brown	239	85
6	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Cu$	Blackish brown	339	80
7	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Zn$	Black	215	87
8	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Co$	Violet	299	88
9	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Ag$	Up white	225	90

3.3 UV- Visible spectral analysis.

Table 3. UV Analysis Data

Sr.no	Complex	Absorption Maxima	Assignment	Geometry
1	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Mn$	220 300	π -π* n-π*	Octahedral
2	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Fe$	250 290	π -π* n-π*	Octahedral

The UV- Visible spectra was recorded in region of 200-800 nm using DMSO [31]. The absorption Maxima at 200 for Mn complex and 250 for Fe complex shows π - π* transition [32]. The electronic absorption spectrum of complex Mn(II) shows absorption Maxima at 220 and 300. And Fe(III) complex shows absorption Maxima at 250 and 290. These charge transfer shows coordination of ligand to metal ion bonding.

3.4 Infrared spectral analysis.

Table 4. IR Spectral Analysis Data

Sr. no	Ligand/Complex	C=N	N-H	Aromatic (C-H)	C=C	M-N	OH
1	$C_{22}H_{14}N_4S_2$	1667	3391	2900	1540	----	----
2	$[(C_{19}H_{21}N_5)(H_2O)_2]Mn$	1611	3332	2965	1535	479	3605
3	$[(C_{19}H_{21}N_5)(H_2O)_2]Fe$	1607	3356	2970	1503	460	3611

The Comparison between the IR spectrum novel Schiff base ligand and its metal complexes determined the changes that might have occurred during complexation. IR spectrum of novel Schiff base ligand exhibit the most characteristic band at 1667 cm^{-1} for V(C=N, azomethine) Vibrational stretching[33-36]. The novel Schiff base ligand showed the band at 3391 cm^{-1} for V(N-H).The band at 1540 cm^{-1} indicates the V(C=C).

Analysis of the Schiff base-Mn complex: The IR spectrum of Mn complex clearly shows the shifting of azomethine band from 1667 cm^{-1} to 1611 cm^{-1}. The band due to imidazole N-H stretching is shifted from 3391cm^{-1} to 3332 cm^{-1}. Also the band of (C=C) stretching vibration shifted from 1540 cm^{-1} to 1535 cm^{-1}. The aromatic C-H stretching vibration in novel Schiff base ligand observed at 2900 cm^{-1} while in metal complex it is observed at 2965 cm^{-1}. The band due to (M-N) stretching vibration observed at 470 cm^{-1} which is absent in the novel Schiff base ligand [37]. This (M-N) stretching vibration band is the characteristic band of metal complex which indicates the linking of nitrogen to the central metal ion in complex formation. The presence of water of crystallization in the complexes is revealed by broad band at between 3605 and 3611 cm^{-1} and result of elemental analysis also support the presence of water of crystallization [38].

Analysis of the Schiff base-Fe complex: The IR spectrum of Schiff base-Fe complex clearly shows shifting of azomethine band from 1667 cm^{-1} to 1607cm^{-1}[39]. The band due to imidazole N-H stretching vibration is shifted 3391 cm^{-1} to 3356 cm^{-1}.The band due to C=C stretching is shifted from 1540 cm^{-1} to 1503 cm^{-1}. The band due to aromatic C-H stretching vibration is shifted from 2900 cm^{-1} to 2970 cm^{-1}.The most characteristic band of M-N stretching vibration is observed at 460 cm^{-1} is absent in the novel Schiff base ligand, which indicates that linking of nitrogen to the central metal ion in complex formation.

3.6 Mass spectral study.

The mass spectrum of the Schiff base shows the molecular ion peak at M/Z 385 (M+1). That corresponds to the molecular weight of the novel Schiff base ligand 384.

3.7 Thermal analysis of metal complexes.

The TGA curve of the metal complex was studied, in which the heating rates were suitably control at 10℃/minute under dynamic nitrogen atmosphere. The weight loss was measured from 28℃ to 500℃. The TGA curve of Mn(II) complex and Fe(III) complex shows total weight loss of 78.60 and 82.70% respectively with the formation of respective metal oxide.

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3.8 Antibacterial activity study.

Table 6. Antibacterial Study Data

Sr. no	Ligand/complexes	Staphylococcus aureus	Salmonella typhi	Aspergillus Niger
1	$(C_{22}H_{14}N_4S_2)$	7.4	9.3	10.2
2	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Ni$	10.3	14.4	10.5
3	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Mn$	8.2	7.5	9.8
4	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Fe$	8.4	8.3	10.6
5	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Cd$	15.5	21.2	22.0
6	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Cu$	7.5	19.6	8.8
7	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Zn$	7.4	13.2	8.7
8	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Co$	9.5	8.8	8.0
9	$[(C_{22}H_{14}N_4S_2)(H_2O)_2]Ag$	11.1	15.8	23.0

The antibacterial activity of novel Schiff base ligand and its metal complexes were studied against gram positive bacteria *staphylococcus aureus* and gram negative bacteria *salmonella typhi* and *Aspergillus Niger* fungi. The antimicrobial activity was carried on in vitro, disc diffusion and agar well diffusion assay [41]. The cultures used were gram positive bacteria *staphylococcus aureus strain NICM 2029. Gram negative bacteria salmonella typhi* strain MTCC 3224 and *Aspergillus Niger* fungi strain NCIM 545. These cultures of microorganism were collected from NICM-National collection of industrial micro-organisms, National chemical laboratory Pune. The microbiological media used for bacteria is nutrient agar and the microbiological media used for fungi is potato dextrose agar. This culture of microorganism was grown for overnight at 37°C [42-43]. The entire investigated compound showed remarkable biological activity against staphylococcus aureus and salmonella typhi in table 7. The results which after obtained reflect that (1) The Cd(II) ,Cu(II), and Ag (II) Complex exhibited very nice antibacterial activity against salmonella typhi showing the zone of inhibition in diameter 21.2 mm, 19.6 mm and 15.8 mm respectively. (2)The Schiff base ligand and complex of Cd(II), Ag(II) and Ni(II) complex shows good antibacterial activity against staphylococcus aureus showing the zone of inhibition in diameter 15.5mm, 11.1mm and 10.3mm respectively. (3) The novel Schiff base ligand and complex of Ag(II), Cd(II) and Fe(III) exhibited excellent antibacterial activity against Aspergillus Niger fungi, showing the zone of inhibition in diameter 23.0mm, 22.0mm and 10.6mm respectively. The result also shows that metal complexes have a high activity against each class of organisms [44]. The comparative study of ligand and the complex is obtained from the novels Schiff base ligand indicates that metal complexes exhibit higher antimicrobial activity. Increased activity of the complexes can be explained on the basis of overtone concept and Tweedy chelation theory [45]. According to the overtone concept of cell permeability, the lipid membrane which surrounding the cell allowed the passage of only liquid soluble materials due to which liposolubility is an important factor controlling the microbial activity. Due to the overlapping of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups the polarity of the metal ion will be reduced to a greater extent on chelation. The next thing is also the mode of action of the compound may involve formation of hydrogen bond through the azomethine group with the active centre of the cell constituent resulting in the interference with normal cell processes.

4 Conclusion

In this research microwave oven is used for synthesis of Schiff base ligand and its metal complexes. This method shows advantage like better yield less reaction time. The synthesized Schiff base ligand and its metal complexes show very nice anti-bacterial activity against staphylococcus aureus, salmonella typhi and Aspergillus Niger fungi.

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