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Microwave assisted solvent free synthesis, characterization and biological activity of Novel Schiff Base ligand and its metal complexes derived from 2-amino-1-methyl benzimidazole and 6-bromo-2-phenyl (4H)-4-benzopyranone

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Abstract

The present work is microwave assisted solvent free, green synthesis of novel Schiff base ligand and its metal complexes. Novel Schiff base ligand was derived from 2-amino-1-methyl Benzimidazole and 6-bromo-2- phenyl (4H)-4-benzopyranone. Metal complexes of Mn(II), Ni(II) Zn(II), Fe(III), Ag(I), Cd(II), Co(II), Cu(II) were synthesized. The novel Schiff base ligand was identified by its color and melting point. Characterization was done by elemental analysis, FT-IR, ^{1}HNMR and Mass spectrometry. Metal complexes were characterized by FT-IR, UV-Visible spectroscopy and TGA analysis. The biological activities were tested against *Staphylococcus aureus, Salmonella typhi and Aspergillus niger.*

Microwave chemistry was introduced in year 1955 and finds a place in one of the green chemistry method. Microwave assisted synthesis is a promising area of modern green chemistry could be adopted to save the Earth from chemical waste[12]. It is solvent free method and best for synthesis of Schiff base ligand and its metal complexes[25,35]. This methodology was time saving, eco-friendly, solvent-free, having simple reaction condition and giving larger yield[3,9,16,19-21,34,36,40]. The reaction of carbonyl compound with primary amine forms Schiff base ligand which contain azomethine (C=N) group[5,22,23]. The metal complexes which are formed from transition metals and Schiff base ligand plays an important role in inorganic chemistry[6]. They have many application in biological and analytical field[13,38,41], such as plant growth inhibitors[28], anticancer[29], anti-tuberculosis, anti-inflammatory and antibacterial[10,27] showing pharmacological activity[18], insecticidal

efficiency[15,33] antibiotics[39]. There is a wide range of applications such as dyes, catalyst, intermediates and stabilizers[17] antiviral[31] and bactericidal properties[34]. Many researchers proved that metal complexes exhibit more potency than metal ligand[8].

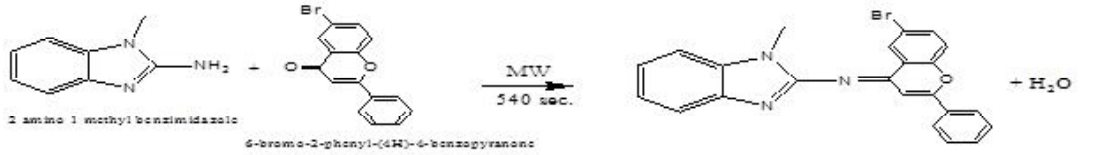
All the chemicals used in this work are of analytical grade. The 2-amino-1-methyl benzimidazole and 6-bromo-2-phenyl-(4H) -4-benzopyranone are from Sigma-Aldrich and metal nitrites and chlorides are from Loba chem and MERCK. The novel Schiff base ligand was synthesized in scientific microwave oven .The metal complexes were synthesized by reacting Schiff base ligand with metal salts in scientific microwave oven.

Techniques :

The novel Schiff base ligand and its metal complexes were synthesized in scientific microwave oven. Digital melting point apparatus was used for measurement the melting point. The elemental analysis was done by in-house technique using Perkin Elmer series ii 2400 CHN/S/O analyzer. Electronic absorption spectra were recorded in the wavelength range 200 to 800 nm using UV spectrophotometer. Simandzu Dr 8031 was used for analysis of IR spectra. The ^{1}HNMR spectra was analyzed in DMSO D6 on Brukers 400 MHz instrument. The Mass spectrum was recorded by LCMS spectrophotometer. The TGA was carried out in dynamic nitrogen atmosphere (30ml/min) with heating rate of 10^{o}C/min. using Shimadzu TGA 50H thermal analyzer.

Preparation of novels Schiff base ligand :

The novel Schiff base ligand was prepared by the reaction between 2-amino-1-methyl benzimidazole with 6-bromo-2-phenyl-(4H) -4-benzopyranone. The reaction was carried out in scientific microwave oven for 9 minutes at 750watt. The fine color change was observed. The microwave irradiated product was cooled at room temperature, and then washed with dry ether and filtered. The final product was recrystallized from absolute ethanol; the yield obtained was 85%. The melting point recorded was 156 . 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 1 methyl benzimidazole

6-bromo-2-phenyl-(4H)-4-benzopyranone

N-(6-bromo-2-phenyl-4*H*-chromen-4-ylidene)-1-methyl-1*H*-benzo[*d*]imidazol-2-amine

Preparation of metal complexes :

The metal complexes were synthesized under solvent-free condition in scientific microwave oven. The metal salts and novel Schiff base ligand were mixed in 1:2 metal-ligand ratios. The reaction mixture was irradiated in scientific microwave oven. The products were washed with ether, filtered and dried at room temperature. The metal salts used were hydrated nickel nitrate, ferric nitrate, cadmium nitrate, copper nitrate, zinc nitrate,

cobalt nitrate, anhydrous silver nitrate and manganese chloride.

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-1-methyl benzimidazole with 6-bromo-2-pheyl (4H) -4-benzopyranone. 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.

Physical properties :

The novel Schiff base ligand and its metal complexes show different colors, which are stable at room temperature; the melting points of ligands show the stability of molecule. The completion time of reaction and high yield proves merits of microwave assisted synthesis. Physical properties of novel Schiff base ligand and metal complexes are shown in table-1.

Elemental composition analysis of novel Schiff base ligand :

Elemental composition analysis data of novel Schiff base ligand is shown in Table-2.

Table-1. Physical properties of novel Schiff base ligand and metal complexes

Sr. No	Ligand/ Complexes	Symbol	Color	M. P. in ^oC	Time (sec.)	Yield (%)
1	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]$	SBL_1	Reddish brown	156	540	85
2	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Ni$	SBL_1Ni	Greenish black	180	120	90
3	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Mn$	SBL_1Mn	Faint orange	281	180	87
4	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Fe$	SBL_1Fe	Brown	250	240	91
5	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Cd$	SBL_1Cd	Violet	229	45	85
6	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Cu$	SBL_1Cu	Dark brown	160	110	90
7	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Zn$	SBL_1Zn	Yellowish	175	50	81
8	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Co$	SBL_1Co	Faint brown	274	60	85
9	$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Ag$	SBL_1Ag	Black	200	210	87

Table-2. Elemental composition analysis of novel Schiff base ligand

Empirical formula	Symbol	Molecular weight	C found (cal.)	H found (cal.)	N found (cal.)	O found (cal.)	Br found (cal.)
$C_{23}H_{17}N_3OBr$	SBL_1	431.13	62.35 (63.35)	4.58 (3.94)	10.58 (9.74)	4.23 (3.72)	18.26 (18.72)

Table-3. Selected frequencies of infrared spectra of novel Schiff base ligand and its complexes

Sr. No	Ligand/ Complexes	υ(C=N) cm^{-1}	υ(N-H) cm^{-1}	υ(C-H) cm^{-1}	υ(C=C) cm^{-1}	υ(M-N) cm^{-1}	υ(C-Br) cm^{-1}
1	SBL_1	1620	3340	3000	1540	----	----
2	SBL_1Cu	1690	3220	2940	1552	460	738
3	SBL_1Ni	1635	3255	3000	1555	480	732
4	SBL_1Zn	1592	3208	2935	1550	495	724
5	SBL_1Co	1635	3206	2999	1513	450	791

From the values of elemental composition analysis of the novel Schiff base ligand it was seen that the percentage of carbon, Hydrogen and nitrogen are in agreement with the proposed structure.

Infrared spectral analysis of novel Schiff base ligand and its metal complexes :

Infrared frequencies of novel Schiff base ligand and metal complexes summarized in Table-3.

The IR spectrum of novels Schiff base ligand shows characteristic band at 1620cm^{-1} which indicates(C=N) stretching vibration of azomethine group[2,4]. The vibrational band at 3340cm^{-1} assigned to N-H stretching in the ligand. The band observed at 1540cm^{-1} correspond to (C=C) stitching. The band at 2975cm^{-1} indicates aromatic C-H stretching in ligand. This confirms the formation of ligand.

IR spectral study of metal complex SBL_1Cu: $[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Cu$

The band appears at 1690cm^{-1} corresponding to an azomethine (C=N) stretching, whereas same band is observed at 1620.39 cm^{-1} in ligand. This indicates coordination of ligand with metal ion[1]. The band appeared at 3000cm^{-1} indicates aromatic C-H stretching in complex. The aromatic C-H stretching was observed at 2975cm^{-1}. The band observed at 3220cm^{-1} assigned to (N-H) stretching where as in spectrum of ligand it is observed at 3366 cm^{-1}. The vibration observed at 1552cm^{-1} due to aromatic (C=C) stretching in metal complex and it is 1540cm^{-1} in ligand. The band at 738cm^{-1} indicates (C-Br) stretching. Band at 3601cm^{-1} in metal complex assigned to $\upsilon(H_2O)$. The characteristics (M-N) vibration seen at the band value 460cm^{-1} due to this the coordination of metal ion and azomethine get confirmed. This band is not appearing in the spectrum of ligand that confirms the formation of metal complex with stable metal ligand bonding. IR spectral study of metal complex of SBL_1Ni: $[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Ni$

Stretching observed at 1635.69cm^{-1} which corresponds to (C=N) stretching vibration. Whereas stretching observed in spectrum of ligand is 1620.39cm^{-1}. The band appeared at 2950cm^{-1} assigned to aromatic (C-H) stretching. Whereas stretching observed in spectrum of

ligand is 2975cm^{-1}. The vibration observed at 1543.91cm^{-1} in complex assigned to aromatic (C=C) stretching. Whereas stretching observed in spectrum of ligand is 1541.31cm^{-1}. The week band at 819cm^{-1} and 730cm^{-1} were due to wagging mode of vibration indicating co-ordination of water molecule in metal complex. The above which are appeared in spectrum of complex are not appeared in the spectrum of ligand confirms the formation of metal complex with stable metal ligand bonding.

IR spectral study of metal complexes of SBL_1Zn: $[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Zn$:

This metal complex shows the azomethine stretching vibration at 1592cm^{-1}. This value is less than the azomethine stretching vibration of novel Schiff base ligand which is at1620cm^{-1}. The (N-H) stretching vibration for complex of Zn observed at 3208 cm^{-1} which is also less than the (N-H) stretching vibration of novel Schiff base ligand which is at 3340cm^{-1}. The (C=C) stretching vibration of carbons of aromatic benzene ring is observed at 1550cm^{-1} for this metal complex but this (C=C) stretching vibration is observed at1540cm^{-1} in novel Schiff base ligand. The Aromatic C-H stretching vibration observed at 2900cm^{-1} for this metal complex but this value is different from the aromatic C-H stretching vibration of novel Schiff base ligand which is observed at 2970cm^{-1}. The metal complex also shows the (C-Br) stretching vibration at 738cm^{-1}. The most characteristic (M-N) stretching vibration of metal complex is observed at 473cm^{-1} which is absent in the novel Schiff base ligand. This value indicates the linking of metal with nitrogen. From this it is confirmed that the formation of metal complex.

IR spectral study of metal complexes of SBL_1Co: $[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Co$:

This metal complex of cobalt shows most characteristic azomethine (C=N) stretching vibration at1635cm^{-1}. This stretching vibration of novel Schiff base ligand observed at 1620cm^{-1}. The (N-H) stretching vibration of metal complex is observed at 3206cm^{-1} while for novel Schiff base ligand this is at 3340cm^{-1}. The (C=C) stretching vibration of aromatic ring is observed at 1513cm^{-1} this is observed at 1540cm^{-1} for the novel Schiff base ligand. The (C-H) stretching vibration of aromatic ring found at 2907cm^{-1} for metal complex this value is 2970cm^{-1} for the novel Schiff base ligand. The (C-Br) stretching vibration observed at 791cm^{-1} for metal complex. The characteristic M-N stretching vibration of metal complex of cobalt observed at the 450cm^{-1} which is absent in the novel Schiff base ligand. From this the formation of metal complex confirmed.

^{1}HNMR spectral studies of novel Schiff base ligands :

^{1}HNMR peaks (ppm) of novel Schiff base ligand are shown in table-4.

Table-4. Observed ^{1}HNMR peaks (ppm) of novel Schiff base ligand

Ligand	H from aromatic ring (ppm)	H from Ar-Br (ppm)	H from N-CH_3 (ppm)
SBL_1	6.86 – 8.12	6.40	3.38

The ^{1}HNMR spectrum of novel Schiff base ligand shows different picks. The characteristic peak exhibited at 3.38 ppm is due to H from N-CH_3. The peak observed at 6.4 ppm is due to H-from Ar-Br. The characteristic peaks exhibited at 6.86 – 8.12 ppm are due to H from aromatic ring.

Mass spectral study :

The fragmentation pattern seen in mass spectrum of novel Schiff base ligand evidently displays molecular ion peak at m/z 431 which equates to the molecular weight 431.13 of the novel Schiff base ligand.

Electronic spectra :

Electronic spectral data and probable geometry for the metal complexes summarized in table-5.

Table-5. Electronic spectral data and probable geometry of the metal complexes

Complex	UV - Vis major bands absorption maxima. cm^{-1} (nm)	Assignment	Proposed geometry
$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Ni$	290	$^3A_{2g} \rightarrow {}^3T_{2g}(F)$	Octahedral
	300	$^3A_{2g} \rightarrow {}^3T_{1g}(F)$	
	410	$^3A_{2g} \rightarrow {}^3T_{1g}(P)$	
$[(C_{23}H_{17}N_3OBr)_2(H_2O)_2]Cu$	275	$^2B_{1g} \rightarrow {}^2A_{1g}$ (υ_1)	Octahedral
	333	$^2B_{1g} \rightarrow {}^2B_{2g}$ (υ_2)	
	409	$^2B_{1g} \rightarrow {}^2E_g$ (υ_3)	

Electronic spectrums of both the metal complexes are recorded in the wavelength region of 200 to 800 nm in DMSO solution.

Electronic spectral data for Ni-complex: Electronic spectrum of Ni(II) complex shows absorption maxima at 290 300 and 400 assigned to $^3A_{2g} \rightarrow {}^3T_{2g}(F)$, $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ and $^3A_{2g} \rightarrow {}^3T_{1g}(P)$ [39-40] transition respectively indicating that complex possesses octahedral geometry.

Electronic spectral data for Cu-complex: Electronic spectrum of Cu(II) complex shows absorption maxima at 275, 333 and 409 assign to $^2B_{1g} \rightarrow {}^2A_{1g}$ (υ_1), $^2B_{1g} \rightarrow {}^2B_{2g}$ (υ_2), and $^2B_{1g} \rightarrow {}^2E_g$ (υ_3) [41] transition respectively indicating that complexes possesses octahedral geometry.

Thermo gravimetric analysis :

The TGA curve of SBL_1Cu was carried out in the temperature range from room temperature to 500°C. The heating was carried out in the nitrogen atmosphere with heating rate 10°C/min. The thermogram of complex shows total weight loss of 82.29% up to 500°C.

From 0°C to 80°C water of crystallization lost with 10% weight loss is observed, loss of organic moiety with total weight loss up to 82.29% at 500°C. A stable curve indicates formation of copper oxide.

The TGA curve of SBL_1 Ni was carried out in the temperature range from room temperature to 500°C. The heating was carried out in the nitrogen atmosphere with heating rate 10°C/min. Thermogram of SBL_1Ni shows weight loss of 80% up to 500°C. From 0°C to 90°C water of crystallization lost with 10% weight loss is observed, lastly loss of organic moiety with total weight loss 80% at 500°C. A stable curve shows the formation of nickel oxide.

Bioactivity study :

The antibacterial activity of novel Schiff base ligand and its metal complexes were summarized in table-6.

Antibacterial activity of synthesized novel base ligand and its metal complexes were performed against gram positive bacteria *Staphylococcus aureus*, Gram negative bacteria *Salmonella typhi* and *Aspergillus niger* fungi. The antimicrobial activities of novel Schiff base ligand and its metal complexes were checked in vitro using disc diffusion method. In this agar diffusion assay disc size 6mm was used. culture used were gram positive Staphylococcus aureus strain NCIM 2079, gram negative Salmonella typhi strain MTCC 3224 and *Aspergillus niger* fungi strain NCIM 545. The culture of microorganism was collected from NICM-National collection of industrial microorganism's National chemical laboratory NCL Pune India. The nutrient agar media microbiological media used for both bacteria and potato dextrose agar media used for fungi. From the table zone inhibition of novel Schiff base ligand and its metal complexes. All the Schiff base ligand and its metal complexes show antibacterial activity against selected

Table-6 Zone of Inhibition of novel Schiff base ligand and its metal complexes

Sr. No	Ligand/ complex	Salmonella typhi (mm)	Staphylococcus aureus(mm)	Aspergillus Niger(mm)
1	SBL_1	7.2	NZ *	8.2
2	SBL_1Ni	17.7	8.2	10.4
3	SBL_1Mn	15.8	10.2	10.5
4	SBL_1Fe	NZ*	7.7	9.8
5	SBL_1Cd	23.6	13.6	17.5
6	SBL_1Cu	16.2	10.4	NZ*
7	SBL_1Zn	11.5	9.3	NZ*
8	SBL_1Co	16.1	16.8	NZ*
9	SBL_1Ag	NZ*	12.9	16.7

bacteria and fungi. The metal complex SBL_1 Cd show excellent antibacterial activity against salmonella typhi bacteria as compared to its parent ligand and other metal complexes. Whereas SBL_1Ni, SBL_1Mn, SBL_1Cu and SBL_1Co are show good antibacterial activity against salmonella typhi bacteria. The complexes SBL_1Cd, SBL_1Co and SBL_1Ag show good antibacterial activity against Staphylococcus aureus bacteria. The complexes SBL_1Cd and SBL_1Ag show good antibacterial activity against *Aspergillus niger* fungi.

In the present work, we developed N-(6-bromo-2-phenyl-4H-chromen-4-ylidene)-1-methyl-1H-benzo[d]imidazol-2-amine and its metal complexes. In this research work synthesis of novel Schiff base ligand and metal complexes were also carried out by using scientific microwave oven. The main advantage of this method is better yield and less reaction time. The time reduces from hours to minutes. It is green and most efficient method of synthesis. This method shows new and simple approach of synthesis. The ligand and its complexes have been characterized by various spectral analyses. Obtained results were in good agreement with the proposed structure. All the metal complexes and novel Schiff base ligand shows good antibacterial activity against Staphylococcus aureus, Salmonella typhi and *Aspergillus niger*.

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