



## Research Article

### SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL SCREENING STUDIES OF SOME METAL COMPLEXES OF NOVEL SCHIFF BASE OF 3-FORMYL-6-METHYLCHROMONE AND 3-AMINOQUINOLINE

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#### ABSTRACT

Synthesis of Ni(II) and Fe(III) complexes with Schiff base 6-methyl-3-((quinolin-3-ylimino) methyl)-4H-chromen-4-one obtained from 3-formyl-6-methylchromone and 3-aminoquinoline. Schiff base and its complexes were characterized by analytical data, elemental analysis, molar conductance, thermogravimetric analysis. The structural investigation performed by UV-Vis, IR and <sup>1</sup>HNMR spectral techniques. Interpretation of elemental analysis shown 2:1 molar ratio of Schiff base with metal complexes. Molar conductance values indicate neutral nature of metal complexes. Antimicrobial activity studied at different concentration (MIC method) by disc diffusion technique. Schiff base and the metal complexes found to be significant antibacterial and antifungal agents.

**Keywords:** Schiff base, antimicrobial activity, 3-aminoquinoline, 3-formyl-6-methylchromone.

#### INTRODUCTION

Schiff bases obtained by the condensation of amino and carbonyl compounds are intrinsic class of ligand that coordinate to metal ions via azomethine nitrogen and have been extensively for its wide range of medicinal applications. Chromone derivatives attract chemist due to various medicinal applications such as antimicrobial, antifungal, antimycobacterial, antiallergenic, antihypertensive, anticonvulsant, anticancer, antioxidant, anti-inflammatory, protein kinase C inhibitors and mushroom tyrosinase inhibition activities <sup>1-5</sup>. Omnipresent occurrence of chromone particularly in plants and their less toxicity towards mammals, also human diet contains huge chromone derivatives <sup>6-7</sup>.

Fluorescence spectra play vital role for further complexation of ligand with metal ion <sup>8-9</sup>. Chemists pay more attention on Schiff base obtained from 3-formylchromone and its derivatives. In the present work, we have synthesized novel ligand from 3-formyl-6-methylchromone and 3-aminoquinoline and its complexes with Ni(II) and Fe(III). Ligand and its complexes are characterized by IR, NMR, UV, elemental analysis. Emphasis has been put on biological evaluation of the complexes.

#### MATERIALS AND METHODS

**Materials:** 3-aminoquinoline, 3-formyl-6-methylchromone, Nickel chloride hexahydrate, ferric chloride hexahydrate and solvent used were AR grade.

**Physical Measurement:** Molar conductance of the complexes was measured in DMF at  $1 \times 10^{-3}$  M using Elico CM-180

conductometer. Elemental analysis (CHN) was carried out using Thermo finnigan, Italy CHN analyzer. Thermal analysis carried out on a Perkin Elmer USAA TGA instrument at heating rate 10 °C/min and temperature range 30°C to 1150°C. The IR spectra ( $4000-400 \text{ cm}^{-1}$ ) in KBr disc were recorded on Bruker, Germany spectrophotometer. The NMR spectra were carried out by mercury plus 300 MHz NMR spectrometer, using TMS as internal standard. The solvent used were chloroform-*d*<sub>6</sub> for Schiff base and DMSO-*d*<sub>6</sub> for metal complexes. Electronic spectra were measured by using Shimadzu UV-160A spectrophotometer. The magnetic moment data obtained by Gouy-type magnetic balance at room temperature using Hg[Co(NCS)<sub>4</sub>] as calibrant.

**In Vitro Antimicrobial Studies:** The antibacterial and antifungal activity of Schiff base ligand and its Ni(II) and Fe(III) complexes towards the bacteria *Klebsiella pneumoniae*, *Staphylococcus aureus* and *Proteus vulgaris* and fungi *Candida albicans* and *Aspergillus niger* was carried out at different concentration by using minimum inhibitory concentration (MIC) method and disc diffusion method. The assay was performed in flat bottom 96 well plate. 1<sup>st</sup> column was used as negative control while second column onward the test drug was added. Initially in second column 2× Muller Hinton broth (100µl) was added while 3<sup>rd</sup> column onwards 1× Muller Hinton broth (100µl) was added. Now 4000 ppm (100µl) concentration of drug was added in second column the drug was mixed properly in order to achieve final concentration of 2000 ppm and now 100µl of solution from second column was taken out and added into 3<sup>rd</sup> column in order to achieve the 2-fold dilution. Finally, 100µl of culture was added to achieve  $1.5 \times 10^6$  cell/ml in each well. Similarly, in the 1<sup>st</sup> row the culture along with diluent and 1× Muller Hinton broth was added. The plates were for incubation and after 24 hours, 5 µl

resazurin (6.75 mg ml<sup>-1</sup>) was added to all wells and incubated at 37°C for another 24 hours. Change of colour was observed and recorded. The lowest concentration prior to colour change was considered as Minimum Inhibitory Concentration (MIC). The culture equivalent to 10<sup>6</sup> cells was added to molten agar and was poured in sterile petri dish and kept for solidification. Wells were made, and samples were added in each well. DMSO and tetracycline was used as negative and positive control respectively. Plates were incubated at 37°C for 24 hours. The zone of clearance was considered for antibacterial activity and was measured in mm.

**Synthesis of Ligand:** The Synthesis of Schiff base (L) was carried out by refluxing hot ethanolic solution of 3-formyl-6-methylchromone (10 mmol, 1.88 gm) in 40 ml of alcohol and 3-aminoquinoline (10 mmol, 1.44 gm) in 15 ml of alcohol. The reaction mixture was refluxed for 5 hours. The progress of reaction was monitored by TLC. The resulting yellow coloured product precipitated, filtered off and washed with ether. The product thus obtained was recrystallized from ethanol.

**Synthesis of the Complexes:** A hot ethanolic solution of ligand (10 mmol, 3.14 gm) was added to ethanolic solution NiCl<sub>2</sub>·6H<sub>2</sub>O (5 mmol, 1.18 gm) and FeCl<sub>2</sub>·6H<sub>2</sub>O (5 mmol 1.35 gm). The resulting reaction mixture was refluxed for 4-5 hours. After cooling, the coloured precipitate obtained was collected, filtered, washed with ether, recrystallized from ethanol and dried in vacuum.

## RESULT AND DISCUSSION

The analytical, physical, molar conductance data and magnetic moment values are shown in table 1. Ligand is chloroform soluble and solubility of metal complexes is in DMSO and DMF. The elemental analysis elucidate that metal to ligand ratio is 1:2 in both Ni(II) and Fe(III) complexes. Neutral nature of metal complexes was proved by the lower value of molar conductivity data.

**IR Spectral data:** IR spectral data are mentioned in table 2. IR spectra revealed,  $\nu(\text{C}=\text{N})$  of band at 1598 cm<sup>-1</sup> is most characteristic band of azomethine, which shifted to 15-20 cm<sup>-1</sup> to lower wave number side in both Ni(II) and Fe(III) complexes<sup>10</sup>. The Schiff base shown a band at 1650 cm<sup>-1</sup>, is assignable to the  $\nu(\text{C}=\text{O})$  group of chromone system. After complex formation the  $\nu(\text{C}=\text{O})$  group is shifted to 6-40 cm<sup>-1</sup> to a lower wave number in its complexes indicate carbonyl oxygen involved in coordination to the metal ion<sup>11-12</sup>. The broad band observed at 3500-3400 cm<sup>-1</sup> and other band at 810-870 cm<sup>-1</sup> that suggest the presence of water molecules in the metal complexes<sup>13-14</sup>. The spectral band observed in the spectral range of 600-400 cm<sup>-1</sup> corresponds to  $\nu(\text{M}-\text{O})$  and  $\nu(\text{M}-\text{N})$  vibrations respectively<sup>15</sup>. Thus the interpretation of IR data suggest that Schiff base ligand (Fig. 1) in reported complexes is bidentate and the coordination of metal ion (Ni(II) and Fe(III)) with Schiff base through azomethine nitrogen and carbonyl oxygen of chromone moiety. The position in the coordination sphere would be completed by water molecule.

**<sup>1</sup>HNMR Spectra:** The <sup>1</sup>HNMR spectrum of ligand recorded in chloroform and its Ni(II) and Fe(III) metal complexes recorded in DMSO. The <sup>1</sup>HNMR data have summarized in table 3. The spectrum of ligand shows following signals: 8.07 ppm (H, S, -HC=N- azomethine proton); 2.35 ppm (3H, S, -CH<sub>3</sub> protons); 6.9-7.9 ppm (m, aromatic protons of chromone nucleus); 7.6-8.9 ppm (m, aromatic protons of quinolone nucleus). However, in metal

complexes the NMR signal of azomethine proton shifted to downfield as compared to NMR signal of azomethine proton in Schiff base<sup>16</sup>. In Ni(II) and Fe(III) complexes resonance signal for azomethine proton at 8.24 ppm and 8.26 ppm respectively. Thus, there is confirming the metal complex formation.

**Electronic Spectra and Magnetic Moment:** The absorption spectra of Schiff base and its Ni(II) and Fe(III) complexes were measured in DMSO solution in wavelength range of 200-1100 nm. The electronic spectra of ligand show two bands, one band at 23310 cm<sup>-1</sup> is attributed to the  $n \rightarrow \pi^*$  transition. Another band at 32258 cm<sup>-1</sup> is due to the  $\pi \rightarrow \pi^*$  transition<sup>8</sup>. The Ni(II) complex showing three bands at 9107 cm<sup>-1</sup>, 15497 cm<sup>-1</sup> and 23696 cm<sup>-1</sup> assignable to the  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$  ( $\nu_1$ ),  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$  ( $\nu_2$ ),  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{P})$  ( $\nu_3$ ) transition respectively characteristic of octahedral geometry<sup>8</sup>. The electronic transition of Fe(III) complex are generally spin forbidden and hence weak, and are often masked by charge transfer spectra<sup>17-18</sup>. The Fe(III) complex showing band at 23148 cm<sup>-1</sup> which can be assigned to the  ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$  transition characteristic of octahedral structure<sup>17-19</sup>. The magnetic moment value of both the complexes as shown in table no. 1 which also confirm their octahedral geometry<sup>8, 18</sup>.

**Antimicrobial Activity:** The antibacterial and antifungal activity of Schiff base and its complexes are shown in the table no 4 and results were compared standard tetracycline. Comparative antibacterial and antifungal studies of Schiff base and its complexes are shown in figure 2. The antimicrobial activity of ligand and its complexes were studied by MIC method<sup>20</sup>. Several gram positive and gram negative bacteria pernicious for human health. The bacteria were mostly found in digestive tract, respiratory tract and skin. The bacterium causes several types of vital organ infections. Due to plasmid bacteria develop resistance in short period of duration. The Schiff base was revealed significant antimicrobial activity against all tested pathogens. The Ligand and Fe(III) complex were shown nearly equal antibacterial activity against all tested organisms when compared with standard antibiotic tetracycline at 2000 ppm. All synthesized compounds were demonstrated better antifungal activity than standard tetracycline at 2000 ppm. There are also other factors which increase the activity are solubility, conductivity, and bond length between metal and ligand<sup>21-23</sup>.

**Thermogravimetric analysis:** Thermogravimetric analysis carried out for [Ni(L)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] and [Fe(L)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] complexes, the range of heating was 30°C to 1150°C and heating rate was 10°C/min. TG curves are shown in figure 3 and 4. Interpretation of thermal data is given in table 5. Nickel complexes, [Ni(L)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] decomposed in two step, in first step complex undergo decomposition with weight loss of 4.63 corresponds to two water molecules in the temperature range between 30°C and 270°C. In the second step, quinolone nucleus with azomethine group undergoes decomposition with weight loss 41.25 in the temperature range of 271°C and 631°C. In the third step complex decomposes gradually with remaining ligand moiety i.e. chromone nucleus and NiO with weight loss 53.37 in the temperature range greater than 631°C<sup>24</sup>. TG studies of Fe(III) complex shown three decomposition step. In the first step decomposition at 30°C to 140°C corresponds to the loss of two water molecules, which are in accordance with the calculated values. The second step of decomposition start from 141°C to 730°C corresponds to chromone nucleus with weight loss 41.72 and in third step temperature greater than 731°C shown decomposition of remaining ligand moiety and FeO residue with final weight loss 52.78<sup>24</sup>.

**Table 1: Physical, analytical, molar conductivity data and magnetic moment of Schiff base and Ni(II) and Fe(III) complexes**

Compounds	Colour (%Yield)	M.P. (°C)	% Found (calcd)				Molar Cond. (ohm <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup> )	$\mu_{\text{eff}}$ (BM)
			C	H	N	M		
L	Yellow (78)	215	76.91 (76.41)	4.06 (4.45)	7.47 (8.90)	—	7	—
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	Green (82)	303	65.80 (66.40)	3.37 (4.42)	7.17 (7.74)	8.20 (8.11)	9	3.12
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	Brown (85)	282	66.43 (66.69)	3.85 (4.44)	6.06 (7.77)	7.53 (7.75)	12	3.41

**Table 2: IR spectral data of ligand and its metal complexes**

Compound	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{O})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
L	1598 cm <sup>-1</sup>	1650 cm <sup>-1</sup>	-	-
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1573 cm <sup>-1</sup>	1641 cm <sup>-1</sup>	490 cm <sup>-1</sup>	552 cm <sup>-1</sup>
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	1572 cm <sup>-1</sup>	1642 cm <sup>-1</sup>	463 cm <sup>-1</sup>	564 cm <sup>-1</sup>

**Table 3: <sup>1</sup>HNMR spectral data of ligand and its metal complexes**

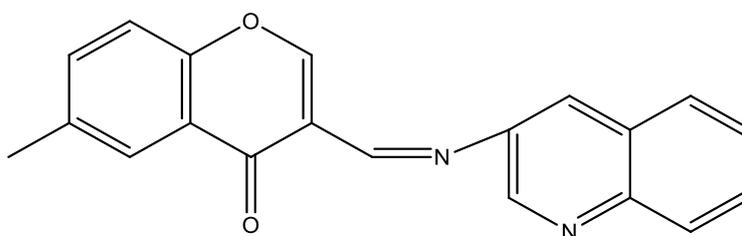
Compounds	Chemical shift (ppm)	Assignment
L	8.07	(H, S, -HC=N- azomethine proton)
	2.35	(3H, S, -CH <sub>3</sub> protons)
	6.9-7.9	(m, aromatic protons of chromone nucleus)
	7.6-8.9	(m, aromatic protons of quinolone nucleus)
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	8.24	(H, S, -HC=N- azomethine proton)
	2.33	(3H, S, -CH <sub>3</sub> protons)
	6.1-7.65	(m, aromatic protons of chromone nucleus)
	7.85-8.85	(m, aromatic protons of quinolone nucleus)
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	8.26	(H, S, -HC=N- azomethine proton)
	2.3	(3H, S, -CH <sub>3</sub> protons)
	6.1-7.8	(m, aromatic protons of chromone nucleus)
	7.9-8.9	(m, aromatic protons of quinolone nucleus)

**Table 4: MIC (ppm) and zone of inhibition values for antimicrobial activity of the ligand and its Ni(II) and Fe(III) complexes**

Microorganism	L diameter of zone of inhibition (mm)				[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ] diameter of zone of inhibition (mm)				[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ] diameter of zone of inhibition (mm)				Standard
	250 ppm	500 ppm	1000 ppm	2000 ppm	250 ppm	500 ppm	1000 ppm	2000 ppm	250 ppm	500 ppm	1000 ppm	2000 ppm	
<i>K. pneumonia</i>	9	12	13	15	3	5	8	11	4	7	10	14	18
<i>S. aureus</i>	8	10	12	16	6	7	10	12	5	8	11	15	16
<i>P. vulgaris</i>	12	14	16	18	4	7	9	13	7	12	15	17	19
<i>C. albicans</i>	10	16	18	20	5	8	11	13	5	8	11	16	14
<i>A. niger</i>	8	10	15	17	9	11	14	16	4	8	12	15	12

**Table 5: Thermal data of complexes**

Complex	Temperature (°C)	Weight loss found (calcd%)	Assignment
[Ni(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	25-270	4.73(4.97)	2H <sub>2</sub> O
	271-630	41.50(42.87)	C <sub>20</sub> H <sub>14</sub> N <sub>4</sub>
	>631	53.77(52.16)	C <sub>20</sub> H <sub>14</sub> O <sub>3</sub> and NiO
[Fe(L) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	30-140	5.50(4.99)	2H <sub>2</sub> O
	141-728	41.72(41.93)	C <sub>20</sub> H <sub>14</sub> O <sub>3</sub>
	>729	52.78(53.08)	C <sub>20</sub> H <sub>14</sub> N <sub>4</sub> and FeO

**Figure 1: Structure of ligand (L)**

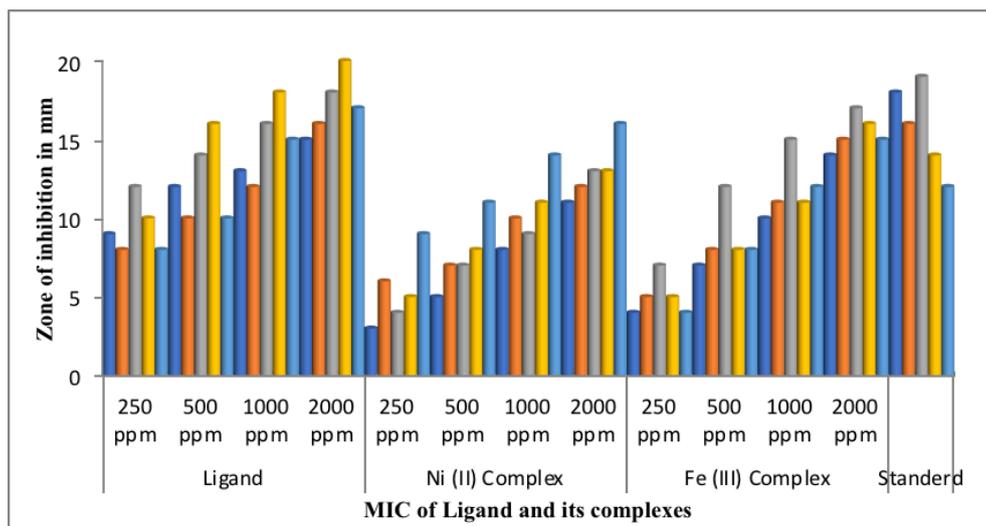


Figure 2: Antimicrobial activity of the ligand and its Ni(II) and Fe(III) complexes

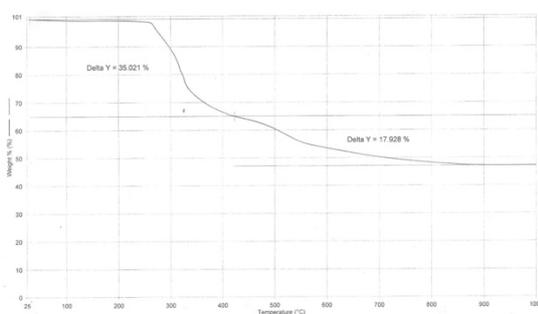


Figure 3: TG graph of (Ni) complex

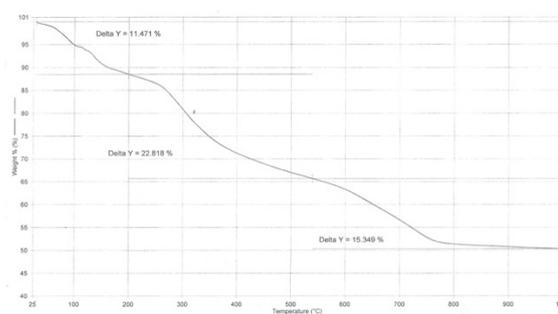


Figure 4: TG graph of Fe(III) complex

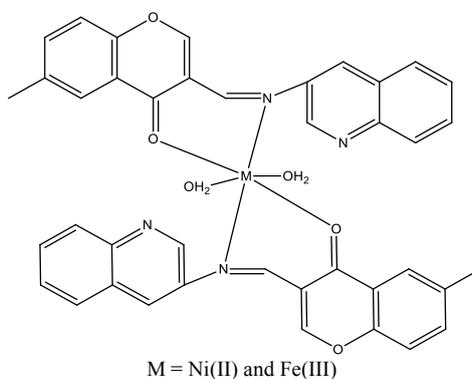


Figure 5: Proposed structure of complexes

## CONCLUSION

Ni(II) and Fe(III) complexes were synthesized from novel Schiff base 3-formyl-6-methylchromone and 3-aminoquinoline and are characterized by using UV-Vis, IR and <sup>1</sup>HNMR spectral techniques. IR interpretation shown that the ligand act as bidentate and metals get coordinated through azomethine nitrogen and carbonyl oxygen of chromone moiety. Octahedral geometry of complexes has been predicted by electronic and magnetic moment data. TGA analysis studies demonstrated the stability of complexes as well as provided the number of

coordinated water molecules. Antimicrobial activity was studied by MIC and disc diffusion method. All prepared compounds were shown good to moderate antimicrobial activity against tested pathogens.

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