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Studies on Ti(III), Cr(III) and Fe(III) complexes with 1-(1-hydroxynaphthalen-2-yl)ethanone-4-chlorobenzoylhydrazone ligand: Synthesis, Physicochemical and Antimicrobial activity

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ABSTRACT

New complexes of 1-(1-hydroxynaphthalen-2-yl)ethanone-4-chlorobenzoylhydrazone (H_2L) with Ti(III), Cr(III) and Fe(III) metal ions have been synthesized and characterized by using various physicochemical techniques such as elemental analysis, molar conductance, diffuse reflectance spectra, magnetic moment, IR, 1H NMR and thermogravimetric analysis (TGA). The analytical data shows formation of 1:1 [metal:ligand] ratio. From spectral data it shows that ligand H_2L is coordinated to metal ions in a tridentate manner through ONO linkage and all complexes exhibit octahedral geometry. Molar conductance values in DMF indicate non-electrolytic behavior of complexes. The Schiff's base H_2L and its metal complexes have been screened for their antimicrobial activities. All the complexes and ligand exhibit more antibacterial activity against bacteria *E. Coli* as compared to standard *penicillin*. While Ti(III) and Fe(III) complexes exhibit good antifungal activity with more than 90% reduction in growth against *A. niger* and *F. moniliformis* respectively.

Keyword: Hydrazone Schiff's bases, transition metal complexes, thermal studies, antimicrobial activity

INTRODUCTION

Hydrazones $-NH-N=CRR'$ (R and R' = H, alkyl, aryl) are versatile ligands and studied widely because they can readily form stable complexes with most of metal ions with interesting coordination mode [1]. The hydrazone Schiff base complexes have been used in catalytic reactions and as models for biological systems. For the past few years, considerable attention has been paid to the chemistry of the metal complexes of Schiff bases containing nitrogen and other donors. This may be attributed to their stability, biological activity such as anti-tumour [2], anti-convulsant [3] and anti-malarial [4]. Hydrazones have potential applications in

many fields such as oxidation catalysis, and electrochemistry [5,6]. Hydrazones also act as herbicides, insecticides, nematocides, rodenticides and plant growth regulators [7].

Literature survey [8-10] reveals that most of study on hydrazone complexes of Ti(III), Cr(III) and Fe(III) was carried out, but the study of metal complexes of hydrazone Schiff base prepared from 4-chlorobenzohydrazide and 2-acetyl-1-naphthol have not been reported so far. The present work aims to synthesize and characterize complexes of 1-(1-hydroxynaphthalen-2-yl)ethanone-4-chlorobenzoylhydrazone (H_2L) with Ti(III), Cr(III) and Fe(III) metal ions. The possible mode

of chelation, the geometry and the nature of bonding of the complexes are discussed based on various spectroscopic methods (diffuse reflectance spectra, IR, ^1H NMR) in addition with thermal study. All the synthesized compounds were screened for antibacterial and antifungal activity.

MATERIALS AND METHODS

All the chemicals and solvents used as starting material for synthesis of ligand and its metal complexes are of analytical grade (AR) and procured from S.D.fine. The metal salts used for preparation of metal complexes are Titanium trichloride (TiCl_3), Chromium chloride hexahydrate ($\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$) and anhydrous ferric chloride (FeCl_3) were commercially available and used as received. 4-chlorobenzohydrazide was synthesized from literature procedure [11]. The metal and chloride contents were determined as per Vogel's procedure [12]. The micro elemental analysis of carbon, hydrogen and nitrogen were done by Elemental Analyser (Thermo Scientific) FLASH- 2000. The infrared spectra of ligands and their metal complexes were recorded on IR Spectrophotometer model RZX (Perkin Elmer) in the $4000\text{-}400\text{ cm}^{-1}$ region using KBr pellets. The diffuse reflectance spectra of the metal complexes were recorded on Jasco UV-Visible Spectrophotometer (V-670) in the range of $1000\text{-}200\text{ nm}$. ^1H NMR spectra of ligands were recorded in DMSO-d_6 on Bruker 400 MHz spectrophotometer, using TMS as an internal standard. Molecular weight measurements were carried out according to Rast's method [13]. The magnetic susceptibility of the metal complexes was measured at room temperature by Gouy's

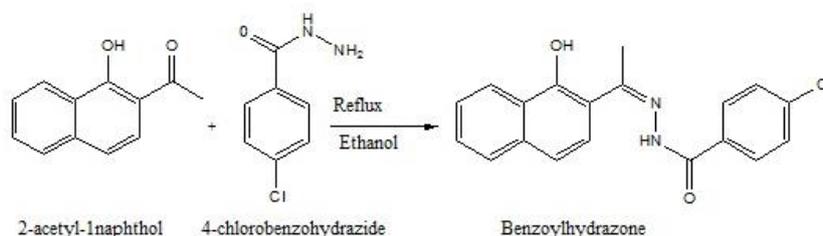
method using mercury (II) tetra thiocyanatocobalt (II), $\text{Hg} [\text{Co}(\text{SCN})_4]$, as a calibrant . Molar conductivity measurements were recorded on a Elico CM-180 conductivity bridge in DMF (10^{-3} M) solution at room temperature. TG analyses of complexes were carried out on TG Instrument, model – SDT Q600 V20.9 Build 20 at a heating rate of 20°C per minute in an atmosphere of nitrogen, within temperature range from room temperature to 800°C .

Preparation of 2-acetyl-1-naphthol by modified Nenchi's method

Freshly fused and powered ZnCl_2 (0.24mol) was dissolved in glacial acetic acid (32ml) in beaker on sand bath. Then add 1-naphthol (0.2mol) with stirring. Heat the reaction mixture on sand bath for 1hr, then cool it and pour it slowly in dil HCl (1:1). Solid product 1-(1-hydroxynaphthalen-2-yl)ethanone obtained was filtered and washed with acidulated water dry it. Product was recrystallized from ethanol [14]. Yield = 92-95%; Melting point = $98\text{-}100^\circ\text{C}$.

Preparation of hydrazone schiff's base

A hot ethanolic solution of 1 - (1 - hydroxynaphthalen - 2 - yl)ethanone was added dropwise to the hot ethanolic solution of 4-chlorobenzohydrazide with constant stirring (scheme-1). The reaction mixture was heated for 3-4 h on waterbath. On cooling to room temperature yellow coloured product obtained was filtered, washed with ethanol and dried under vacume of CaCl_2 [15]. Finally recrystallized from DMF-ethanol mixture (1:4v/v). Yield = 72%; Melting point = $200\text{-}201^\circ\text{C}$.



Scheme 1: Synthesis of hydrazone schiff's base (H_2L)

Preparation of metal complexes

To a hot DMF solution (15ml) of organic ligand (0.1mol), a hot ethanolic solution of the

appropriate metal salt (0.1mol) solution was added under continuous stirring. The mixture was refluxed on sand bath for 6-8h. The

resultant solution was digested to half of its volume; on cooling solid product was obtained. The product was washed with ethanol followed by petroleum ether and dried at room temperature [16]. Yield = 65-70%.

Biological evaluation

Antimicrobial study of synthesized Schiff base H_2L and its Ti(III), Cr(III) and Fe(III) complexes are studied as:

Antibacterial activity

Bacterial cultures used:

- i) Gram positive: *Staphylococcus aureus*, *Bacillus subtilis*
- ii) Gram negative: *Escherichia coli*, *Salmonella typhi*

Standard used: *Penicillin*

Method used: Agar Cup Method

Nutrient agar (Hi media) was prepared and sterilized at 15 psi for 15 min. in the autoclave. It was allowed to cool below 45°C and seeded with turbid suspension of test bacteria separately, prepared from 24 hrs old slant cultures, 3% inocula were used everytime. This seeded preparation was then poured in sterile petri plate under aseptic condition and allowed to solidify. Cups of 10mm diameter were bored in the agar plate with sterile cork borer. 100µl of compound solution prepared in DMSO (1%) was added in the cup under aseptic condition with the help of micropipette. 100µl of DMSO was also placed in one of cup as blank (negative control). Then the plates were shifted to incubator at 37°C and incubated for 24hrs. Results were recorded by measuring the zone of inhibition in mm using zone reader [17].

Antifungal activity

Fungal cultures used: *Aspergillus niger*, *Penicillium chrysogenum*, *Fusarium moneliforme*, *Aspergillus flavus*.

Standard used: *Gresiofulvin*

Method used: Poison plate method

The medium used was Potato Dextrose Agar (Hi media). The medium was prepared and sterilized at 10 psi in autoclave for 15min. Then the

compound to be tested is added to the sterile medium in aseptic condition so as to get final concentration as 1%. A plate with DMSO was prepared as blank (negative control). All the selected test fungal cultures were allowed to grow on slant for 48 hrs so as to get profuse sporulation. 5ml of 1:100 aqueous solution of Tween 80 was added to the slant and spores were scraped with the help of nicrome wire loop to form suspension. The plates were incubated at room temperature for 48hrs. After inoculation plates were observed for the growth of inoculated fungi. Results were recorded as growth of fungi (No antifungal Activity), reduced growth of fungi (Moderate antifungal Activity) and no growth of inoculated fungi (Antifungal activity) [17].

RESULTS AND DISCUSSION

The ligand and complexes synthesized are colored, stable towards air and moisture at room temperature. The complexes are generally insoluble in water, slightly soluble in ethanol and methanol, while completely soluble in DMF and DMSO solvent. The analytical data of the ligand and complexes are given in Table 1, which confirm $C_{19}H_{15}ClN_2O_2$ molecular formula of ligand and 1:1 metal to ligand stoichiometry in complexes. The molar conductance values of the complexes in DMF at $10^{-3}M$ are found to be in between range of 9.8 - 20 $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$ was indicate the non electrolytic nature of the complexes [18].

1H NMR study of ligand H_2L

The structure of ligand can be confirmed from 1H NMR spectra as shown in Fig.1 recorded in DMSO- d_6 and data is presented in Table 2. The 1H NMR spectrum (in δ ppm) of ligand H_2L displayed a singlet observed at 14.82 ppm (S, 1H, -OH) was assign to phenolic -OH group. Another singlet observed at 11.34 ppm (S, 1H, imino) [19] was assign to proton of imino -NH group. The three protons of methyl group have resonated as a sharp singlet at 2.55 ppm (S, 3H, -CH₃) [20]. Signals in the region 8.28-7.40 ppm are assigned for aromatic protons.

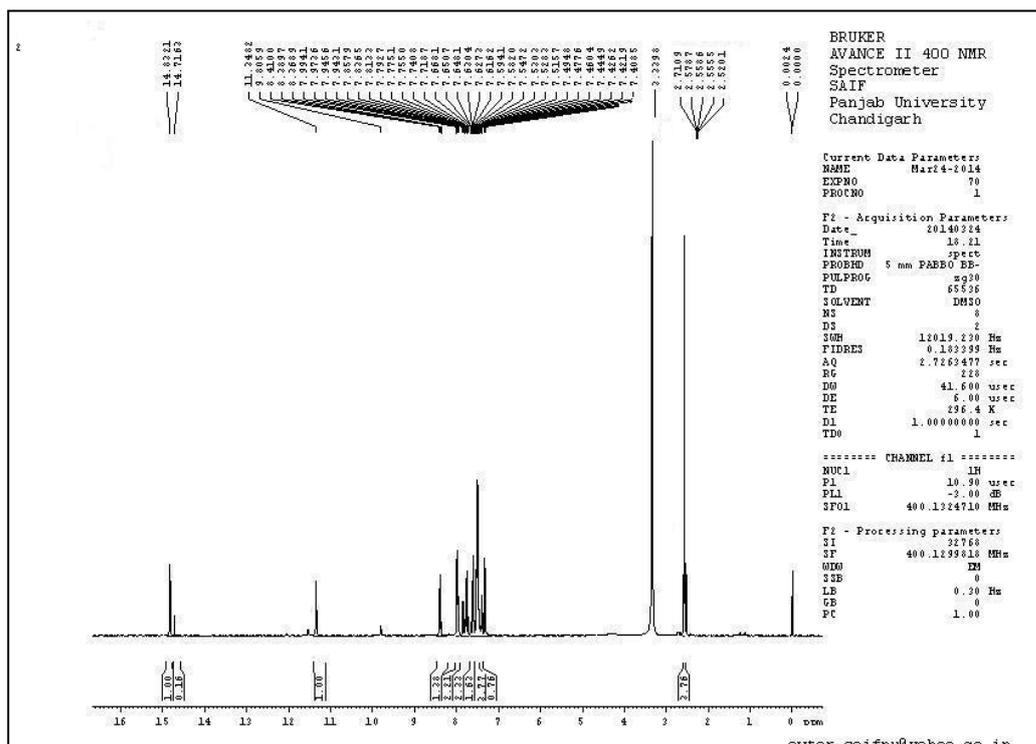
Table 1: Analytical and Physico-Chemical data of the Schiff's base ligand (H₂L¹) and its Metal Complexes

Molecular Formula Ligand/ Complexes	Molecular weight	Colour	Elemental Analysis %found (cal.)					μ^{eff} (BM)	Λ M*
			C%	H%	N%	M%	Cl%		
C ₁₉ H ₁₅ ClN ₂ O ₂ (H ₂ L)	338.78	Yellow	67.03 (67.36)	4.40 (4.46)	8.18 (8.27)	—	10.38 (10.46)	—	—
[Ti(L)Cl ₂ .H ₂ O]	474.56	Cherry	46.00 (46.09)	3.34 (3.40)	5.76 (5.90)	10.00 (10.09)	22.34 (22.41)	1.60	22
[Cr(L)Cl ₂ .H ₂ O]	478.69	Chocolate	47.56 (47.67)	3.27 (3.37)	5.49 (5.85)	10.38 (10.86)	22.08 (22.22)	3.76	20
[Fe(L)Cl ₂ .H ₂ O]	482.54	Seaweed	47.17 (47.29)	3.14 (3.34)	5.65 (5.81)	11.46 (11.57)	22.00 (22.04)	5.90	18

* Molar conductance values in $\text{ohm}^{-1} \text{cm}^2 \text{mole}^{-1}$.

Table 2: ¹H NMR spectral data of ligand H₂L

Compound	Chemical shift δ ppm	Peak and no. of proton	Type of proton
Ligand (H ₂ L)	δ 14.82	1H (S)	-OH (phenolic)
	δ 11.34	1H (S)	-NH (imino)
	δ 8.28 – δ 7.40	10H (m)	Ar-H
	δ 2.55	3H (S)	-CH ₃

**Fig. 1 ¹H NMR spectra of ligand H₂L**

Magnetic moment and Diffuse reflectance spectra

The proposed structure of synthesized Ti(III), Cr(III) and Fe(III) complexes were studied from the recorded diffuse reflectance spectra and

spectral data was presented in Table 3. Magnetic moment value of Ti (III) complex was found to be 1.60 B.M. and shows one broad band at 18903 cm^{-1} assigned to ${}^2T_{2g} \rightarrow {}^2E_g$ transition, confirms octahedral geometry of complex. The

magnetic moment value for Cr (III) and Fe(III) complex was found to be 3.76 and 5.90 B.M. respectively [21, 22]. Electronic spectra of Cr (III) complex shows three transitions at 16051, 23640 and 33003 cm^{-1} assignable to ${}^4\text{A}_{2g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$, ${}^4\text{A}_{2g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{F})$ and ${}^4\text{A}_{2g}(\text{F}) \rightarrow {}^4\text{T}_{1g}$

(P) transitions respectively and for Fe(III) complex shows three transition at 15337, 19607 and 25316 assignable to ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}(\text{G})$, ${}^6\text{A}_{1g} \rightarrow {}^6\text{T}_{1g}(\text{G})$ and ${}^6\text{A}_{1g} \rightarrow {}^4\text{E}_g$, ${}^4\text{A}_{1g}(\text{G})$ respectively, suggesting octahedral geometry.

Table 3: Diffuse reflectance spectral data of complexes

	Absorption bands (λ_{max})		Assignment
	nm	cm^{-1}	
[Ti(L)Cl ₂ .H ₂ O]	529	18903	$2\text{T}_{2g} \rightarrow 2\text{E}_g$
[Cr(L)Cl ₂ .H ₂ O]	623	16051	$4\text{A}_{2g} \rightarrow 4\text{T}_{2g}$
	423	23640	(F)
	303	33003	$4\text{A}_{2g} \rightarrow 4\text{T}_{1g}$
			(F)
			$4\text{A}_{2g} \rightarrow 4\text{T}_{1g}$
			(P)
[Fe(L)Cl ₂ .H ₂ O]	652	15337	$6\text{A}_{1g} \rightarrow$
	510	19607	$4\text{T}_{1g}(\text{G})$
	395	25316	$6\text{A}_{1g} \rightarrow$
		$6\text{T}_{1g}(\text{G})$	
		$6\text{A}_{1g} \rightarrow 4\text{E}_g$,	
		$4\text{A}_{1g}(\text{G})$	

IR Spectral studies

The modes of bonding in complexes are compared with free ligand, from study of IR spectral data given in Table 4 and in Fig 2. IR bands for $\nu(\text{N-H})$, $\nu(\text{C=O})$, $\nu(\text{C=N})$ and phenolic $\nu(\text{C-O})$ are observed at 3211, 1633, 1593 and 1286 cm^{-1} respectively in free ligand. However in the o-hydroxy Schiff base a medium broad band at 3005 cm^{-1} was observed due to the presence of a strong intramolecular hydrogen bonding (O-H...N=C). In complexes this band was disappeared which indicates the coordination of phenolic oxygen to metal ion via deprotonation which was confirmed by upward shift of phenolic $\nu(\text{C-O})$ frequency 1292-1324 cm^{-1} compared to ligand with frequency 1286 cm^{-1} [23]. Stretching

frequency of $\nu(\text{C=N})$ in free ligand shift to lower value by 4-23 cm^{-1} in complexes [24]. This indicates coordination of azomethine nitrogen to metal ion in complexes. Presence of $\nu(\text{C=O})$ and $\nu(\text{N-H})$ vibrations in the spectra of Ti(III), Cr(III) and Fe(III) complexes indicate that hydrazone ligand was in keto form. This confirmed monobasic tridentate nature of ligand in Ti(III), Cr(III) and Fe(III) complexes. All complexes show bands in region at 3400-3350 cm^{-1} , 1618-1633 cm^{-1} and 729-742 cm^{-1} assignable to $\nu(\text{OH})$, $\delta(\text{H}_2\text{O})$ and $\rho_w(\text{H}_2\text{O})$ modes, respectively [25]. The bands observed in the far-infrared regions at 518-570 and 422-493 cm^{-1} are assignable to the $\nu(\text{M-O})$ and $\nu(\text{M-N})$ vibrations, respectively.

Table 4: Infrared spectral data of ligand and its complexes (cm^{-1})

Ligand/ Complexes	$\nu(\text{OH})+$ $\nu(\text{NH})$	$\nu(\text{NH})$	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{N-N})$	$\nu(\text{C-O})$ phe	$\nu(\text{M-O})$	$\nu(\text{M-N})$
H ₂ L	3005	3211	1633	1593	979	1286	---	---
Ti(III)	---	3196	1625	1587	1014	1324	553	422
Cr(III)	---	3194	1631	1570	1010	1292	584	449
Fe(III)	---	3198	1624	1589	1008	1311	518	493

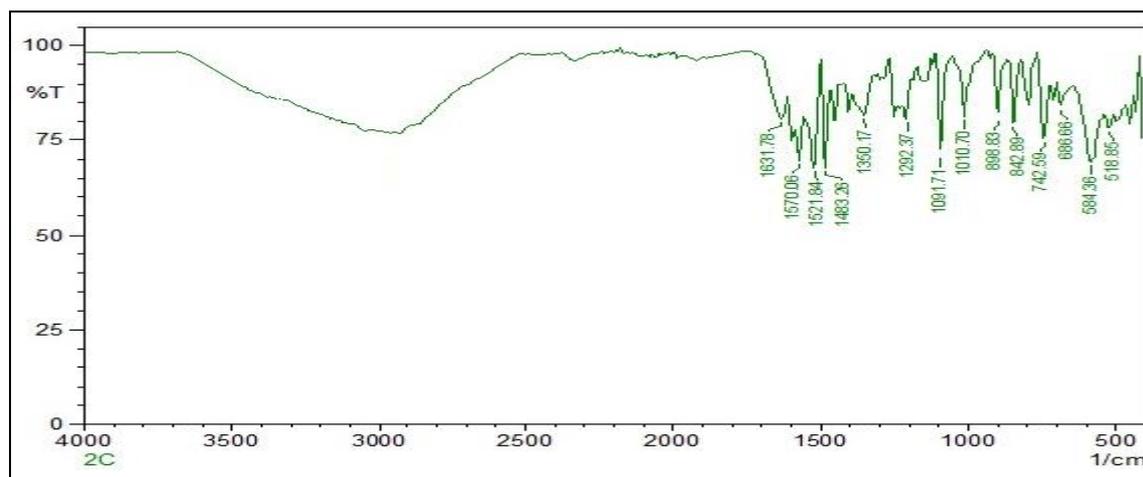
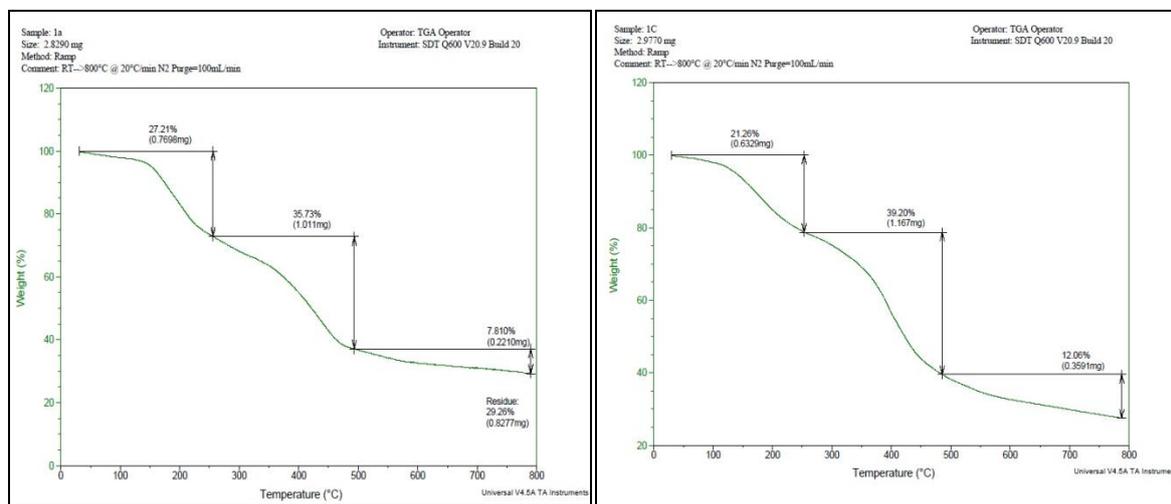


Fig. 2 IR spectra of $[\text{Cr}(\text{L})\text{Cl}_2\cdot\text{H}_2\text{O}]$ complex

Thermogravimetric analysis

Thermal behaviors of metal complexes were investigated by TGA technique. The thermogram of metal complexes presented in Fig. 3 indicates that the complexes Ti(III), Cr(III) and Fe(III) shows a three stage decomposition pattern. In case of Ti(III), Cr(III) and Fe(III) complexes decomposition begins at 150°C , 145°C and 140°C with weight loss 27.21%, 21.26% and 25.09% corresponds to one coordinated water molecule and two chloride ions and some decomposition part of ligand respectively. The second step for Ti(III), Cr(III) and Fe(III) complexes with

35.73%, 39.20% and 40.60% weight loss, in the temperature range $245\text{--}500^\circ\text{C}$, $250\text{--}480^\circ\text{C}$ and $200\text{--}510^\circ\text{C}$ respectively, represent partial decomposition of ligand moiety. Further decomposition of remaining part of ligand was continued upto 800°C with observed weight loss 7.810% for Ti(III) complex, 12.06% for Cr(III) complex and 9.81% for Fe(III) complex. High residue for Ti(III), Cr(III) and Fe(III) complexes was due to some organic part and metal oxide, indicate strong coordination bond between metal and ligand [26].



[A]

[B]

Fig. 3 Thermogram of complexes [A] $[\text{Ti}(\text{L})\text{Cl}_2\cdot\text{H}_2\text{O}]$ and [B] $[\text{Cr}(\text{L})\text{Cl}_2\cdot\text{H}_2\text{O}]$

Antimicrobial study

Antimicrobial data for ligand and its complexes was presented in Table 5. Compared to standard used and its metal complexes, ligand H_2L was

less active towards all microorganisms. This is because of the chelation according to Tweedy's chelation theory [8].

Table 5: Antibacterial (mm) and antifungal activity of ligand H₂L and its metal complex

Compounds	Antibacterial activity (mm)				Antifungal activity			
	Gram -ve		Gram +ve		A. niger	P.chryso genum	F.moneli forme	A. flavus
	E. coli	S. typhi	S. aureus	B. subtilius				
H ₂ L	12	n.a.	12	10	RG	+ve	RG	RG
Ti(III)	16	n.a.	15	13	-ve	RG	RG	RG
Cr(III)	13	12	15	14	RG	RG	RG	RG
Fe(III)	14	13	14	12	RG	RG	-ve	RG
Penicillin	11	24	36	30	-	-	-	-
Griseofulvin	-	-	-	-	-ve	-ve	-ve	-ve

n. a. - No activity

+ve - Growth (Antifungal Activity absent)

-ve - No Growth (More than 90% reduction in growth, Antifungal Activity present)

RG - Reduced Growth (More than 50% and less than 90% reduction in growth)

Antibacterial activity

All metal complexes are moderately active against all bacterial stains. Ti(III) (16mm), Cr(III) (13mm) and Fe(III) (14mm) exhibits high activity against E. coli as compared to standard Pencillin (11mm). Ti(III) complex was inactive towards S. typhi .

Antifungal activity

Ti(III) complex exhibits good antifungal activity against A. niger. In case of Fe(III) complex, F. moneliforms possess more than 90% reduction in growth. Cr(III) complex shows reduced growth against all fungal strains.

CONCLUSION

The bonding of ligand to metal was confirmed by the analytical, IR, electronic, magnetic and thermal studies. From the present investigation it has been observed that Schiff base H₂L was monobasic tridentate towards Ti(III), Cr(III) and Fe(III). Analytical and physicochemical studies reveals that the octahedral geometry was assigned for Ti(III), Cr(III) and Fe(III) complexes. The metal complexes have higher antimicrobial activity than the ligand. Generally ligand and its complexes both exhibits higher activity against E. Coli bacteria than standard Pencillin.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this paper.

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