



Synthesis, Characterization and antimicrobial Screening of 1-((E)-[(5-chloro-2-hydroxyphenyl)imino]methyl)-2-naphthol Metal complexes

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ABSTRACT

Abstract: The Schiff's base of 2-hydroxy naphthaldehyde with mono chloro aminophenol have been synthesized by simple condensation reaction. Further the Schiff's base ligands were made complexes with different metals like copper, cobalt, nickel and manganese. The newly synthesized complex molecules were characterized by IR, and UV spectral analysis. To all the complexes, the antimicrobial activity was evaluated.

1. Introduction

Coordination chemistry is an important branch of inorganic chemistry since the appearance of water on earth aqua complex ion of metal must have existed¹. Schiff bases have often been used as chelating ligands in coordination chemistry²⁻³, they are more effective as a chelating agent when they bear supporting and stabilizing group like -OH close to -HC=N- group. Bidentate group containing imine groups have also been used as the modulators of structure and electronic properties of transition metal centers. The ligand Schiff base finds its applications as the analytical reagent for determination of metals⁴⁻⁵. Transition metal complexes containing the Schiff base ligands have been interest for many years⁶⁻⁸. The compounds encompassing -C=N-(Azomethine) group are known as Schiff bases, which are usually synthesized by the condensation of primary amines and active carbonyl groups. Schiff bases are important class of compounds in medicinal and pharmaceutical field. They are well known in pharmaceutical industry and have been shown to possess a broad spectrum of biological activities. In light of these significances, varieties of synthetic strategies have been developed for the preparation of Schiff base. They have been found to exhibit the pharmacological activities⁹ such as antimalarial¹⁰, anticancer¹¹, antibacterial¹², antifungal¹³, antitubercular, anti-inflammatory¹⁴, antimicrobial¹⁵, antiviral¹⁶ etc. They also serve as a back bone for the synthesis of various heterocyclic compounds. In view of the above biological importance of Schiff bases, we have planned to synthesize some novel benzoxazole analogs of Schiff bases in this chapter.

In recent days, chemists and industrialists are thinking towards green chemistry. Synthetic methodologies have been designed to use and to generate substances that possess little or no toxic effect to human health and to the environment. Schiff bases belong to this class of compounds and widely used group of organic intermediates, which are important for the production of rare chemicals, e.g. pharmaceuticals or rubber additives¹⁷ and as amino protective groups in organic synthesis¹⁸⁻¹⁹. They are also used as liquid crystals²⁰ and in analytical,²¹ medicinal²² and polymer chemistry²³.

Complex's introduction: The use of N-heterocyclic carbenes (NHCs) as ligands is now ubiquitous in modern organometallic chemistry and there are examples of NHC complexes with a large number of the transition metals²⁴⁻²⁶. Of particular interest is the catalytic behavior exhibited by NHC complexes of palladium, ruthenium and rhodium, and more recently nickel, platinum and iridium. NHC complexes of these metals catalyze a broad range of reactions including C-C and C-N coupling, olefin metathesis, polymerization and hydrogenation.

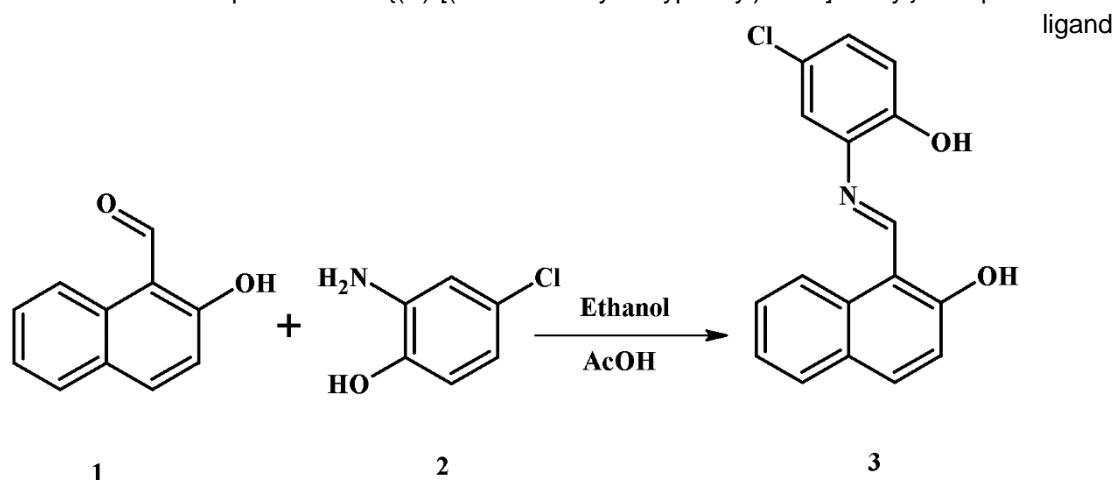
By keeping these literatures as background, we have synthesized novel complexes which contain organic ligands reacts with inorganic metals and also the target molecules were evaluated for antimicrobial activities.

Present work

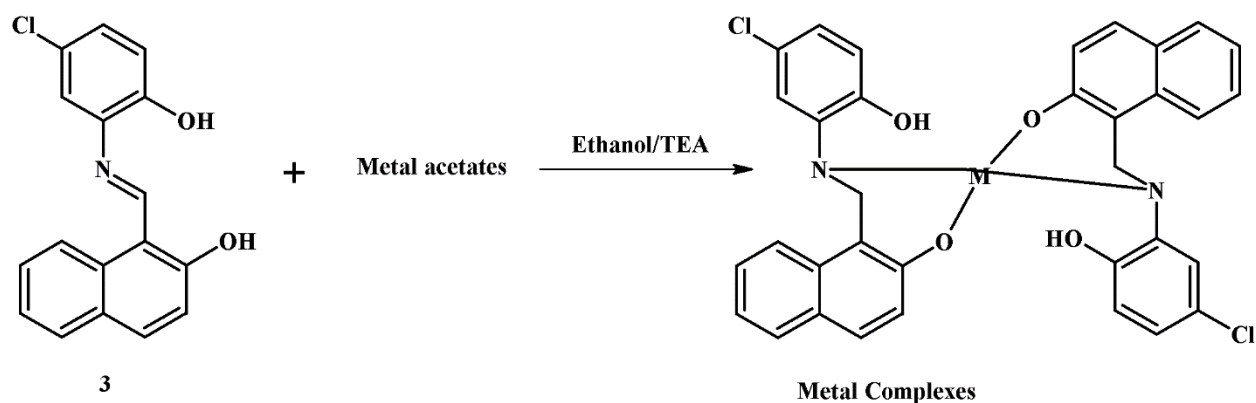
Encouraged by the literature reports and to further assess the pharmacological profile of metal complexes of heterocyclic systems, it was thought worthwhile to construct the Schiff's base metal complexes, which may impart enhanced biological activity to the resulting compounds and that we have to be check. The complexes further screened for antimicrobial activity.

The synthetic strategy involved the following steps,

- Preparation of 1-((E)-[(5-chloro-2-hydroxyphenyl)imino]methyl)-2-naphthol **3**
- Preparation of Metal complexes of 1-((E)-[(5-chloro-2-hydroxyphenyl)imino]methyl)-2-naphthol Schiff base ligand



Scheme-1. Synthetic route for Schiff Base Ligand



Scheme-2. Metal complexes Synthesis

$M = \text{Co, Cu, Mn, Ni}$

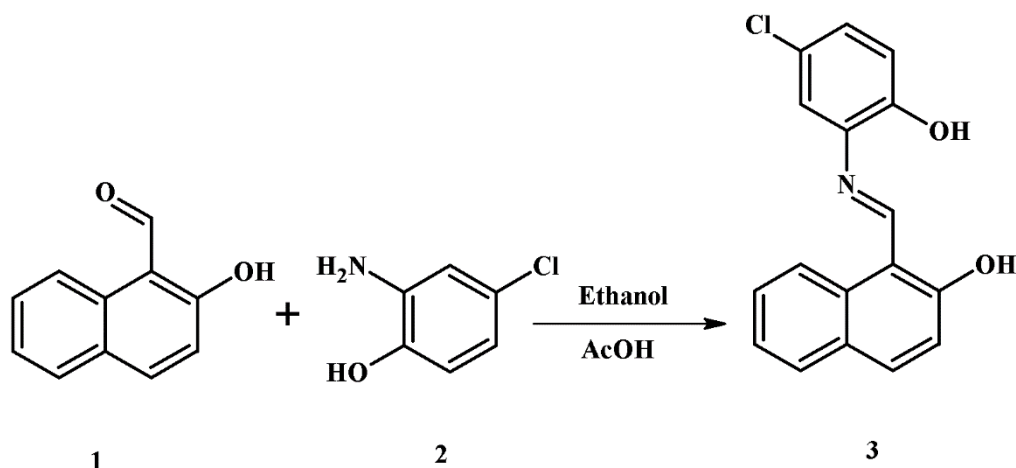
2. Experimental section

2.1 Chemistry

Melting points were recorded on electro thermal melting point apparatus and are uncorrected. ¹H NMR and ¹³C NMR spectra were recorded on Bruker 400 MHz spectrometer. Chemical shifts were shown in δ values (ppm) with tetramethylsilane (TMS) as an internal standard. LC-MS were obtained using C18 column on Shimadzu, LCMS 2010A, Japan. The FT-IR spectra of compounds were taken in KBr pellets (100 mg) using Shimadzu Fourier transformed infrared (FT-IR) spectrophotometer. Column chromatography was performed using silica gel (60-120 mesh). Silica gel GF254 plates from Merck were used for TLC and the spots were observed by UV light. The chemicals were purchased from Sigma Aldrich Co. and solvents for column chromatography were of reagent grade and purchased from commercial source.

- Preparation of 1-((E)-[(5-chloro-2-hydroxyphenyl)imino]methyl)-2-naphthol **3**

2-hydroxy-1-naphthaldehyde (0.01 mole) **1**, monochloro aminophenol **2** (0.01 mole) and catalytic amount of acetic acid were taken in ethanol (50 ml) and refluxed for 3-4 h. The completion of the reaction was monitored by TLC. Then the reaction mass was cooled and filtered to get solid product **3**.

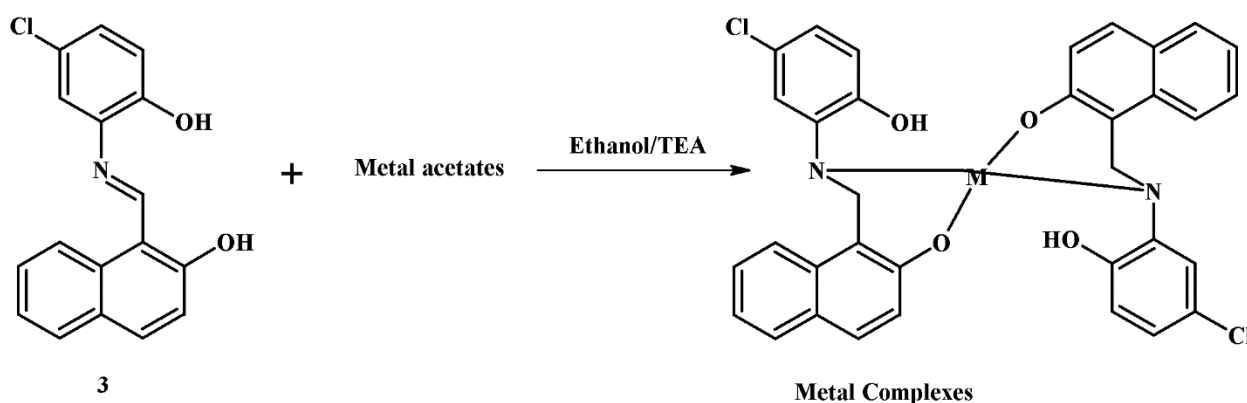


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The obtained ligand was characterized by IR, ¹HNMR and Mass spectral analysis and confirmed the formation of product. The ¹HNMR (Fig. 7) signal for –HC=N (Schiff base) appeared at the region of 11.2 δ value. For the hydroxyl protons the peak appeared at the region of 8.9 and 10.3 δ value. The 9 aromatic protons exhibit signals at 7.1-8.7 δ value. In IR analysis the strong absorbance peak at the region of 3400 cm⁻¹ supports the formation of target molecule.

- i. Preparation of Metal complexes of 1-((E)-[(5-chloro-2-hydroxyphenyl)imino]methyl)-2-naphthol Schiff base ligand

The Schiff base ligand 3 (0.001 mole) and different metal acetates taken in round bottom flask and dissolved in Ethanol. The catalytic amount of Tri-ethylamine also added to reaction mass. The reaction mass is refluxed for about 7-8 hr and allowed for cooling to room temperature. The obtained solid was filtered, dried and recrystallized using ethanol.



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The metal complexes of transition metals Cu, Co, Mn and Ni have been prepared using Schiff base ligand 3. The obtained products have been subjected to IR and UV-visible spectral analysis. The ligand shows the UV absorption at 320nm wavelength and the respective metal complexes exhibit 2 peaks absorption i.e. one for the ligand and another for the metal present in the complexes. The IR spectra's of metal complexes also shown strong absorption peak at 3480 cm⁻¹ confirms the presence of –OH group, which is not involved in the formation of complexes. The spectra's have been plotted below.

Spectra's

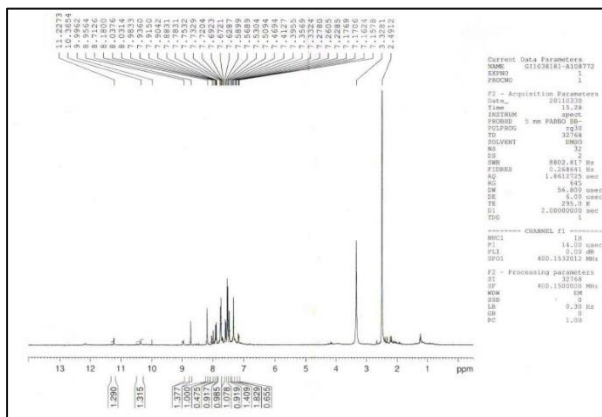


Fig.1 ¹H NMR spectra of Schiff's base Ligand

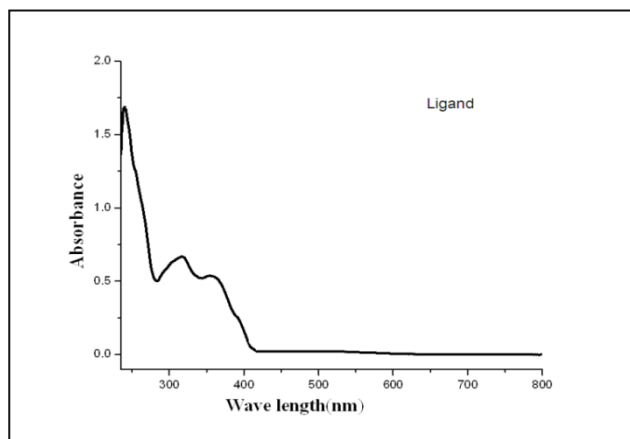


Fig.2 UV spectra of Schiff's base Ligand

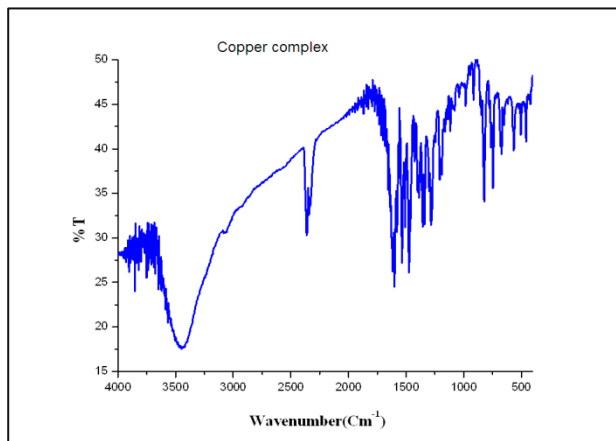


Fig. 3. IR spectra of Copper Complex

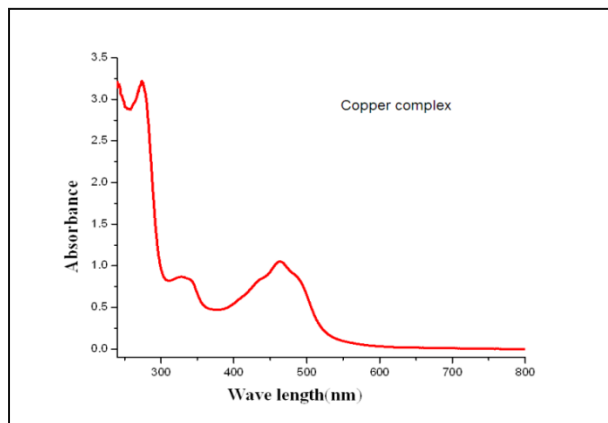


Fig. 4. UV spectra of Copper Complex

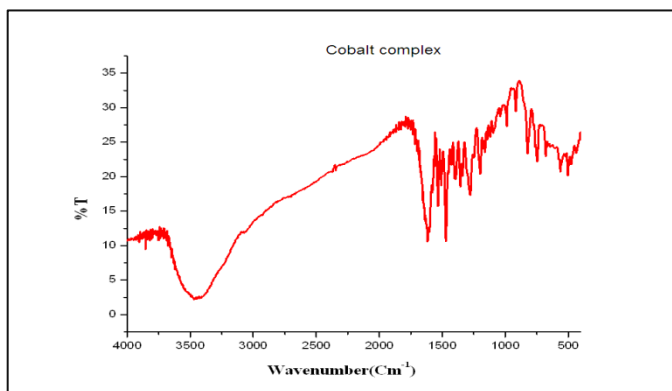


Fig. 5. IR spectra of Cobalt Complex

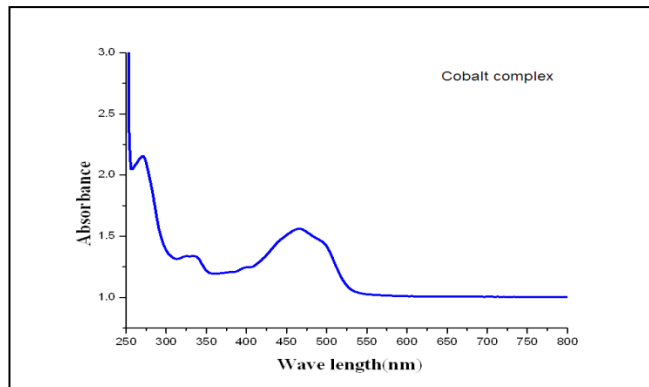


Fig. 6. UV spectra of Cobalt Complex

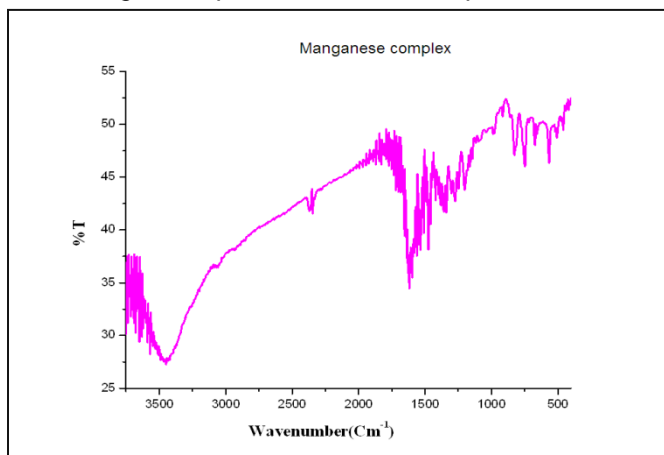


Fig. 7. IR spectra of Manganese Complex

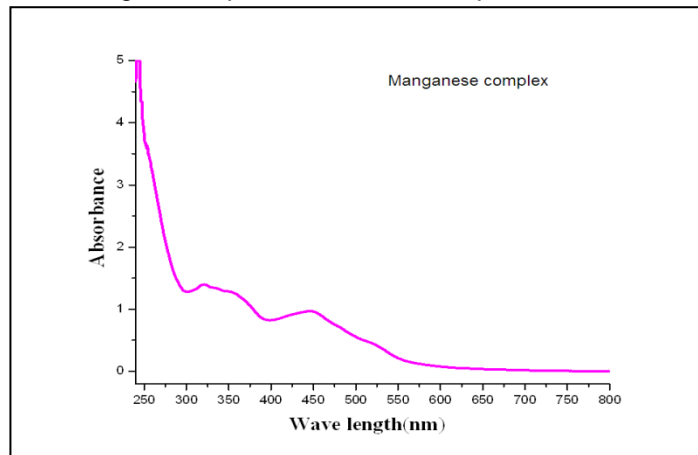


Fig. 8. IR spectra of Manganese Complex

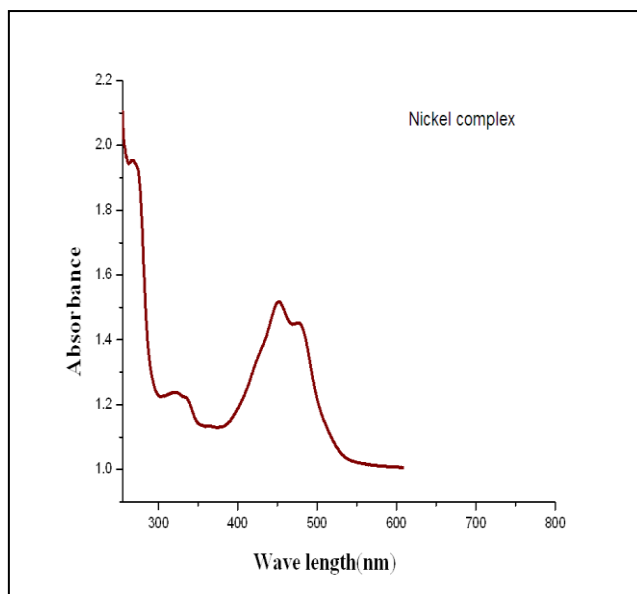


Fig 9. UV spectra of Nickel Complex

3. Antimicrobial activity

The antimicrobial activity of the synthesized metal complexes was carried out using agar well diffusion method⁶⁷. The bacterial strains were collected from patients with different infectious status who had not administered any antibacterial drugs for at least two weeks with the suggestions of an authorized physician in Kiran diagnostic health Centre of Chitradurga, Karnataka state, India. Fungal strains were procured from the culture maintained at National College of Pharmacy Shimoga. The In vitro antimicrobial activity was carried out against 24 h culture of ten bacterial strains, such as *S. pyogenes*, *Bacillus subtilis*, *S. epidermidis*, *B. cereus*, *Streptococcus* (Gram positive), *K. pneumoniae*, *P. aeruginosa*, *V. cholerae*, *E. aerogens*, *S. flexneri* (Gram negative). The four fungal strains like *C. neoformans*, *Trichosporan*, *A. flavus* and *Candida albicans* were used to determine antifungal activity. The compounds were tested at 40 µg/mL concentration against both bacterial and fungal strains. DMSO was used as vehicle. Ciprofloxacin (40 µg /100 µL) and Fluconazole (40 µg /100 µL) were used as standard drugs for the comparison of antibacterial and antifungal activities respectively. The zone of inhibition was compared with standard drug after 24 h of incubation at 37°C for antibacterial activity and 72 h at 25°C for antifungal activity. All the tested compounds were considered to be equipotent since the values obtained were very close to each other. The compounds 1 and 2 exhibited higher antimicrobial activity when compared to other compounds with respect to the standard drug followed by the complex 4. The antibacterial results were recorded in Table-1. The results of antifungal activity were recorded in Table-2.

Table-1
Antibacterial activity of metal complexes

Comp	Zone of Inhibition in cm									
	Gram +ve Bacteria					Gram -ve Bacteria				
	<i>B. subtilis</i>	<i>S. pyogenes</i>	<i>S. epidermidis</i>	<i>B. cereus</i>	<i>Streptococcus</i>	<i>K. pneumoniae</i>	<i>P. aeruginosa</i>	<i>V. cholerae</i>	<i>E. aerogens</i>	<i>S. flexneri</i>
1	2.7	1.3	1.6	3.0	1.5	2.0	2.2	1.0	1.0	3.0
2	1.6	1.0	1.0	1.2	1.2	1.0	1.5	00	1.5	1.5
3	1.4	00	00	1.8	00	00	1.3	1.0	00	1.0
4	1.6	00	00	1.1	00	0.8	1.6	00	00	0.6
DMSO	00	00	00	00	00	00	00	00	00	00
Std.	3.1	2.8	2.9	3.2	2.6	3.0	3.1	2.9	2.7	3.2

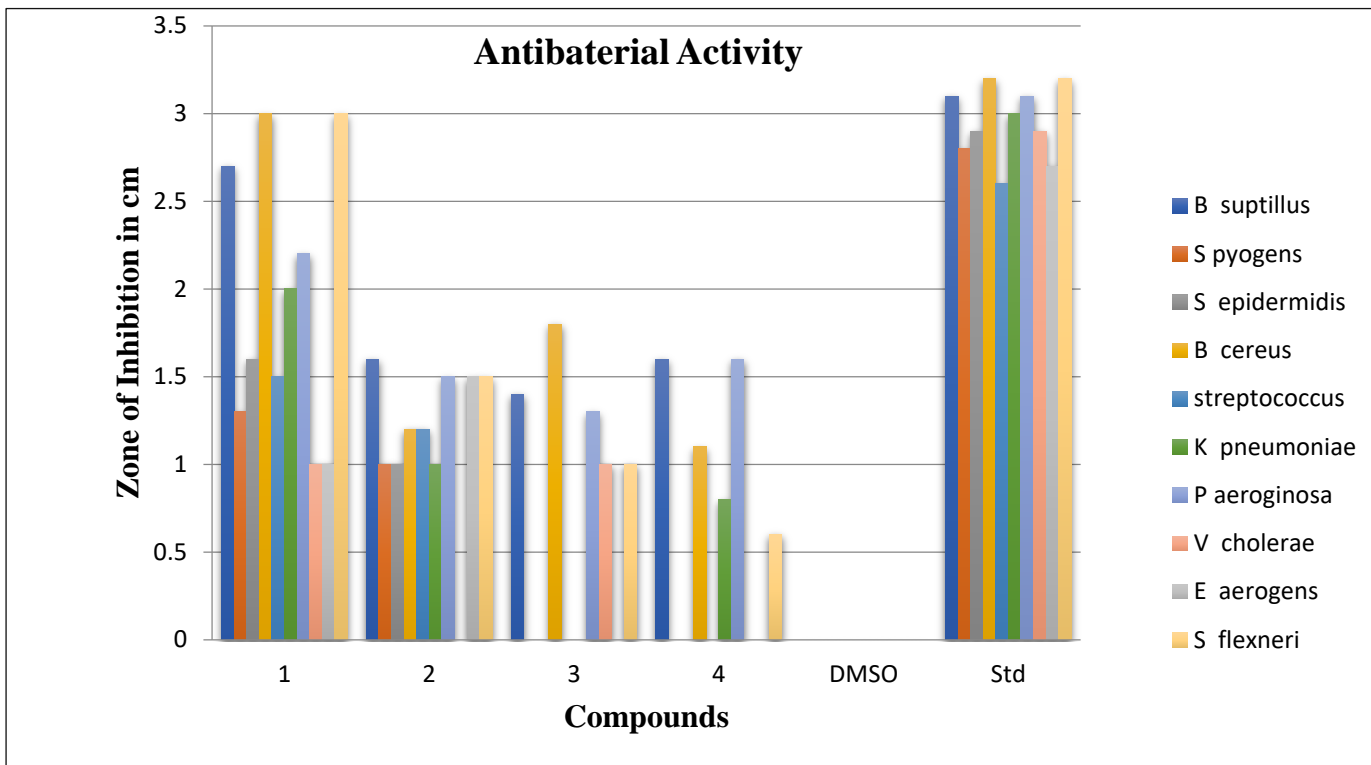


Fig.10 Antibacterial activity of metal complexes

Table-2
Antifungal activity of metal complexes

Complexes	Zone of Inhibition in cm			
	<i>C albicans</i>	<i>C neoformens</i>	<i>Trichosporan</i>	<i>A flaves</i>
1	2.5	1.0	2.5	2.6
2	1.6	0.0	2.0	2.0
3	1.1	0.0	1.8	2.1
4	1.9	0.0	1.5	1.5
DMSO	0.0	0.0	0.0	0.0
Standard	3.0	2.8	3.1	3.0

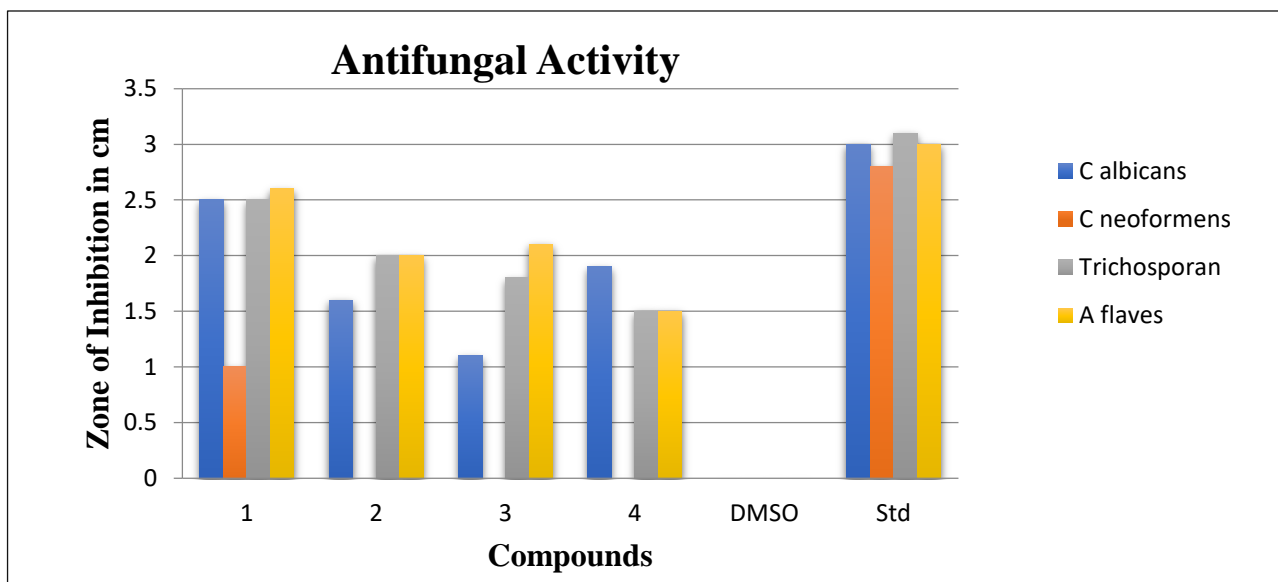


Fig.11 Antifungal activity of metal complexes

Table-3
Physical data of the synthesized compounds

Compounds	Molecular Formula	Molecular Weight	M.P.(°C)	Yield (%)
Ligand	C ₁₇ H ₁₂ ClNO ₂	297.7	199	85
Copper complex	[C ₃₄ H ₂₄ Cl ₂ N ₂ O ₄]Cu	659.01	Decomposed	90
Cobalt complex	[C ₃₄ H ₂₄ Cl ₂ N ₂ O ₄]Co	654.4	265	92
Manganese complex	[C ₃₄ H ₂₄ Cl ₂ N ₂ O ₄]Mn	650.4	203	87
Nickel complex	[C ₃₄ H ₂₄ Cl ₂ N ₂ O ₄]Ni	654.16	249	95

4. Conclusion

In the present work, Schiff's base of 2-hydroxy naphthaldehyde with mono chloro aminophenol has been synthesized. The obtained Schiff base has been characterized by IR, ¹H NMR and Mass spectroscopic analysis. The Schiff base further used to synthesis metal complexes. The complex of Cu, Co, Mn and Ni have been synthesized and characterized by UV-Visible and IR spectroscopy. The targeted complexes subjected to screen for antimicrobial activity. The Cu and Co complexes exhibited prominent antimicrobial activity when compared with other complexes. Considering all the data's obtained, it can be concluded that the synthesis of some metal complexes via simple method has been achieved and the spectral analysis supports the formation of final molecules.

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