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# Synthesis, Spectral Studies and Antimicrobial Activities of *P*-Anisalidene-*O*-Aminophenol and Their Metal Complexes

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## ABSTRACT

The complexes of Mn(II), Fe(II), Co(II), Ni(II) and Cu(II) were synthesized from the complexation reaction between the Schiff base ligand and their metal ions. The Schiff base ligand, *p*-anisalidene-*o*-aminophenol (ANSAP) were derived by the condensation reaction of 2-aminophenol with *p*-anisaldehyde. All the compounds were characterized by elemental analyses, Molar conductance, FT-IR, UV-Visible, $^1$H-NMR. The data from spectral studies revealed that the Schiff base act as a neutral bidentate ligand. The complexes were tested out for antibacterial activities against *Xanthomoans* sp. and *Staphylococcus aureus VTCC BAA 20*. Antifungal activity against *Fusarium oxysporum* sp. *lycoperici* and *Candida albicans* sp. was also carried out using agar well diffusion method. It was reported that all the metal complexes were more potent than the parent ligand (ANSAP).

**Keywords** *p*-anisaldehyde, 2-aminophenol, Metal complexes, Antibacterial activity, Antifungal activity.

## INTRODUCTION

Schiff bases are easily prepared and form complexes nearly with all metal ions. Several Schiff base complexes show significant catalytic activity in different reactions at very high temperature (>100 ◦C) and in the presence of moisture (Gupta *et al*. 2008). Schiff bases are growing importance in present days due to their broad applications in different fields (Kajal *et al*. 2013). The class of compounds, so-called imines, are often referred to as Schiff bases, named after the German chemist Hugo Schiff (Zhang *et al*. 2018). Schiff bases are synthesized by the condensation reaction of carbonyl functional groups with primary amines (Cimerman *et al*. 2000). Structurally, Schiff bases are nitrogen analogue of an ketone or aldehyde in which carbonyl group is displaced by an azomethine (-HC=N-) and imine (-C=N-) groups (Silva *et al*. 2010). The process of Schiff base preparation normally takes place under acid or base catalysis with heat (Antony *et al*. 2016). The primary amine uses to synthesize Schiff base, can be an aliphatic or aromatic amine. These compounds are often called as imines, azomethines or anils (Ashraf *et al*. 2011). The aldehydes or ketones used to synthesize Schiff base may be aromatic or aliphatic. Although Schiff base ligands with ketones formed is less commonly

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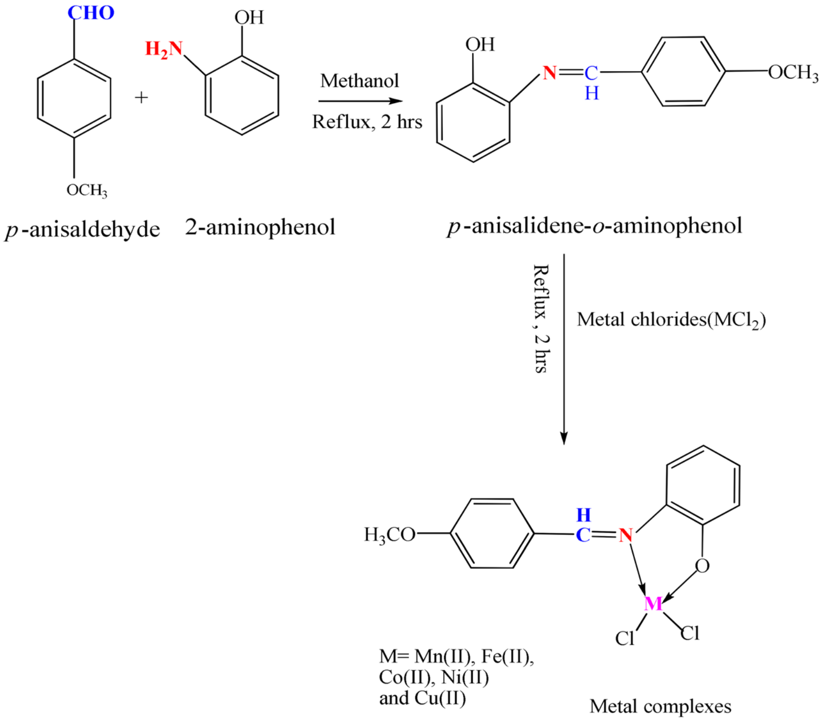
than aldehydes (Kumar *et al*. 2009). It is reported that anisaldehyde derivatives with one or more O, N atoms in the aromatic ring and its complexes, shows biological activities like antibacterial, antitumor and antifungal activities (Ekinchan *et al*. 2002). During the past years, metal-based drugs gained more importance in medicinal field (Buldurun *et al*. 2019).

Schiff bases have ability to coordinate with many metals (Cozzi 2004) and stabilize them in different oxidation states (Wahab *et al*. 2003) to control their activites for broad applications (Nair *et al*. 2006). The transition metals have an incomplete d and f shell in their neutral or cationic states. These incomplete valency shell orbital act as Lewis acid and accept the lone pair from Lewis base, like Schiff Base (*N*, *O* and *S* donor ligand) (Sunand *et al*. 2010). In plants, transition metal ions like Mn, Zn, Cu, Co and Fe are found as micronutrients and are essential for their life cycle and for various activities. The transition metal complexes have been nitrogen and oxygen donor. Some complexes of metals have attractive chemical and physical characteristics (Omar *et al*. 2009) because they show different colors and highly stable for biological activities (Ghanghas *et al*. 2021). Binding studies of metal complexes have become an important field in the development of chemotherapeutics and DNA molecule probes in upcoming year (Kalarani *et al*. 2020). Schiff bases consists structural lability, sensitive to molecular environment and unusal configuration (Wahab *et al*. 2003).

Schiff bases are one of the most broadly used Organic compounds (More *et al*. 2019). Azomethine or Imine groups are present in different natural, non-natural and natural derived compounds. The imine group present here shows to be analytic for their biological activities (Silva *et al*. 2010). It is believed that the biological activity is related to the hydrogen bonding through the imino group of Schiff bases with the active centers of the cell constituents. Schiff base also acts as catalysts (Garg and Kumar 2003), dyes, pigments, intermediates in organic synthesis, corrosion inhibitors and polymer stabilizers (Kumar 2016). Schiff base obtained from amino compounds possess a wide range of applications in clinical, biochemical,



**Scheme 1.** Synthesis of *p*-anisalidene-*o*-aminophenol and its metal complexes.

analytical (Mounika 2010), biological and pharmalogical areas (You *et al*. 2008). Schiff bases and its metal complexes described to show a broad range of biological activities like antibacterial including antifungal (Mezey *et al*. 2016), anti-HIV (Joseyphus and Nair 2008), antiparasitic, antiviral, antimalarial, anti-inflammatory, antioxidant, pesticidal (Ding *et al*. 2014), cytotoxic, enzyme inhibitory, anticancer including DNA damage (Emam *et al*. 2018), antiulcer activity (Parashar *et al*. 1988). There are several Schiff bases and their complexes which gained more attention in various fields involving synthesis of optical (both and fluorescent and colorimetric) molecular probes, supramolecular materials, magnetic materials, cell imaging and organic light emitting diodes (Cheng *et al*. 2014).

## MATERIAL AND METHODS

The chemicals and reagents were collected from Merck, HiMedia and CDH of analytical grade (AR) which were used in the experiment with high purity, without any additional processing. Schiff base ligand were synthesized by the condensation of *p*-anisaldehyde with 2-aminophenol and its co-ordination behavior with different metal complexes were studied (Scheme 1). Infrared spectra were recorded in KBr pellets in the region 4000-400 cm$^{-1}$. The solution was prepared in ethanol to obtain the UV-Visible spectra of complexes in the range of 200-800 nm. Proton nuclear magnetic resonance spectra of complexes are recorded in DMSO using TMS as an Internal references. The Schiff base and its metal complexes were also tested for their antimicrobial activity.

## RESULTS AND DISCUSSION

Newly synthesized metal complexes are crystalline solid having different colors. The ligand and their metal complexes are generally soluble in common polar organic solvents such as DMSO, chloroform, DMF, ethanol and methanol (Akbar ali *et al*. 1977).

### Elemental analyses

Element analyses like Nitrogen, Hydrogen, Carbon and Chlorine of newly synthesized Schiff base ligands and its metal complexes has been achieved by the Volhard′s Method (Vogel 1975) (Table 1).

### Conductance studies

Molar conductance of all the metal complexes (1a-1e) at *ca*. 1× 10$^{-3}$ M at 25 ºC were measured in DMSO. All the data of molar conductance ($\Lambda_m$) values for the complexes are compiled in Table 1. Molar conductance of all the complexes shows low values (15-28 S cm$^2$ mol$^{-1}$) telling that they are non-electrolytic in

**Table 1.** Physical, analytical data and molar conductance for ANSAP and its metal complexes.

| Sl. No. | Compounds | Empirical formula (Formula weight) | M.pt. (°C) | Color (Yield %) | Calculated (Found) (%) | | | *Molar conductance (Λm) S cm$^2$ mol$^{-1}$ |
|---|---|---|---|---|---|---|---|---|
| | | | | | C | H | N | |
| 1 | ANSAP | $C_{16}H_{19}NO_2$ (257.14) | 81-83 | Yellow (85) | 74.68 (74.53) | 7.44 (7.32) | 5.44 (5.30) | - |
| 1a | [Mn(ANSAP)Cl$_2$] | $C_{16}H_{18}Cl_2MnNO_2$ (381.01) | 150-151 | Dark brown (70) | 50.29 (50.07) | 4.75 (4.60) | 3.67 (3.03) | 16.78 |
| 1b | [Fe(ANSAP)Cl$_2$] | $C_{16}H_{18}Cl_2FeNO_2$ (382.01) | 158-160 | Black (75) | 50.17 (50.08) | 4.74 (4.58) | 3.66 (3.49) | 15.76 |
| 1c | [Co(ANSAP)Cl$_2$] | $C_{16}H_{18}Cl_2CoNO_2$ (385.00) | 145-148 | Light brown (85) | 49.77 (47.65) | 4.70 (4.72) | 3.63 (3.50) | 19.74 |
| 1d | [Ni(ANSAP)Cl$_2$] | $C_{16}H_{18}Cl_2NiNO_2$ (384.01) | 168-170 | Brown (85) | 49.80 (48.95) | 4.70 (4.63) | 3.63 (3.49) | 25.22 |
| 1e | [Cu(ANSAP)Cl$_2$] | $C_{16}H_{18}Cl_2CuNO_2$ (389.00) | 135-137 | Black (80) | 49.18 (49.02) | 4.64 (4.47) | 3.58 (3.42) | 27.32 |

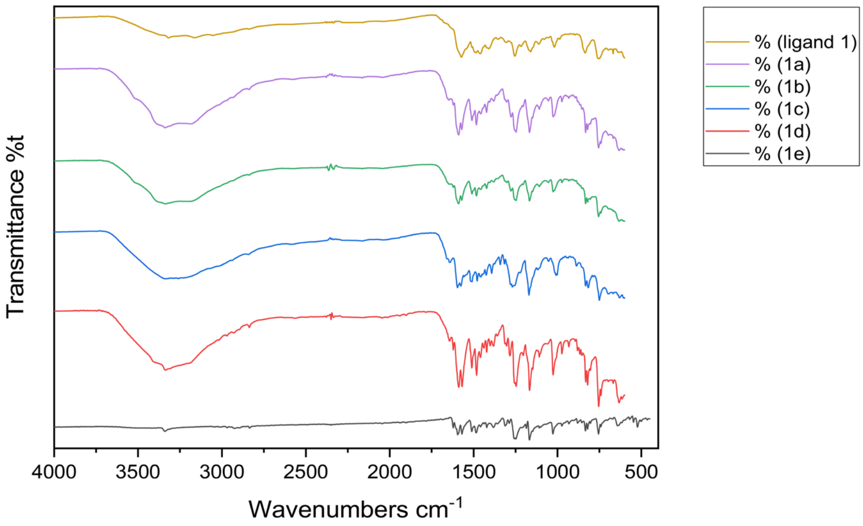


**Fig. 1.** IR spectra of ANSAP and its complexes.

nature (Chohan *et al.* 2001), (Geary 1971, Greenwood *et al.*1968).

## FT-IR spectra

The IR data for Schiff base and its metal complexes are given in Table 2 and shown in Fig. 1.The IR spectrum (Fig. 1) of the free ligand (ANSAP) showed characteristic bands at 1488 cm$^{-1}$ and 1622 cm$^{-1}$ due to ν(C=O) and ν(C=N) respectively (Mart 2004). Due to the ν(C=O) stretching vibration of the Schiff base ligand, the band 1488 cm$^{-1}$ shifts to lower frequencies 1467-1484 cm$^{-1}$ in the metal complexes confirming the coordination of the carbonyl oxygen (C=O) to the metal ions. Another stretching frequency in free ligand due to phenolic group ν(C-OH) is observed at 3005 cm$^{-1}$ and the absence of these spectral bands in all metal complexes indicated the bond formation between phenolic-oxygen to metalions. In the spectra of the complexes, the presence of bands in the region of 768-781 cm$^{-1}$ were due to ν(M–O) stretching vibration which was absent in the spectra of free ligand. In the spectra of the complexes, the azomethine band at 1622 cm$^{-1}$ shifts to lower frequencies ranging to 1596–1609 cm$^{-1}$ due to coordination of azomethine nitrogen atom to the metal ions. In the IR spectra of these complexes, the new bands is observed in the region of 608-615 cm$^{-1}$ because of the ν(M–N) vibrations but these bands is not observed in the spectra of free ligand which indicates that there is no complexation (Nath and Chaudhary 1998).

**Table 2.** IR Data (cm$^{-1}$) for ANSAP and its metal complexes.

| Complex | ν(C-OH) | ν(C=N) | ν(C=O) | ν(M-N) | ν(M-O) |
|---|---|---|---|---|---|
| 1 | 3005 | 1622 | 1488 | - | - |
| 1a | - | 1609 | 1476 | 615 | 769 |
| 1b | - | 1597 | 1479 | 610 | 773 |
| 1c | - | 1596 | 1483 | 611 | 768 |
| 1d | - | 1597 | 1484 | 608 | 770 |
| 1e | - | 1587 | 1467 | 614 | 781 |

## UV-Visible spectra

In the spectra (Fig. 2) of the ligand (ANSAP) shows band at of 228 nm which is attributed to π-π* transition of the benzene ring of the ligand, where this

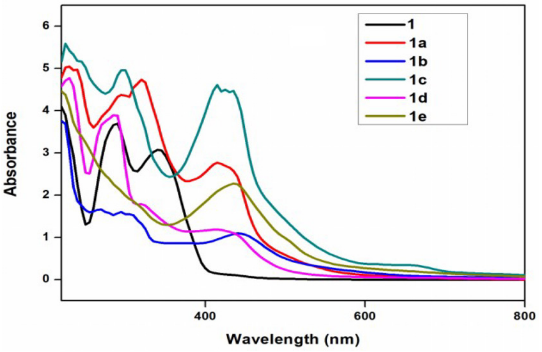


**Fig. 2.** UV-Visible spectra of ANSAP and its complexes.

**Table 3.** UV-Visible data for Schiff base, ANSAP and its metals complexes.

| Complex | Benzenoid $\pi - \pi^*$ | Azomethinechromophore | |
|---|---|---|---|
| | | $\pi - \pi^*$ | $n - \pi^*$ |
| 1 | 228 | 240 | 318 |
| 1a | 230 | 248 | 303 |
| 1b | 241 | 259 | 307 |
| 1c | 249 | 257 | 306 |
| 1d | 230 | 262 | 302 |
| 1e | 232 | 253 | 308 |

band is slightly changed in the spectra of complexes. The absorption spectra of the free ligand shows a band at 240 nm that attributed to the $\pi$ -$\pi^*$ transitions of the azomethine group (Mart 2004). During the formation of the complexes, this band is shifted to higher wavelength (red shift). Furthermore, another electronic spectral band seen at 318 nm indicating the n-$\pi^*$ transitions of the azomethine chromophore, on complexation this weak band is shifted to lower wavelength called as blue shift(Nath and Chaudhary 1998). This blue shift suggests that the nitrogen atom of the azomethine group is coordinated to the metal ion (Table 3).

### $^1$H NMR spectra

A comparison of the spectrum (Figs. 3 - 4) of the free ligand with their complex in freshly synthesized DMSO solution is given in Table 4. In the $^1$H NMR spectra of Schiff base ligand, a singlet peak appeared at 8.62 δ ppm was assigned due to protons of azomethine group. In the metal complexes, there was a downfield shift of this peak in the range of 9.73-9.81 δ ppm indicating the coordination of azomethine nitrogen to metal ion. The aromatic protons also displayed multilet peaks in the range of 6.81-7.96 δ ppm. The phenolic OH group assigned a broad singlet at 8.88 δ ppm in spectra of the Schiff base ligand. In the metal complexes, this broad singlet was disappeared confirming the absence of OH phenolic proton and involvement of the oxygen atom in complexation. In the spectra of free ligand, the signal for methoxy group (C-OCH$_3$) attached to anisaldehyde ring show at the 3.84 δ ppm, these signal was shift to upfield in metal complexes in the range of 3.68-3.80 δ ppm.

**Table 4.** $^1$H NMR data (δ ppm) for ANSAP and its metal complexes.

| Complex | Protons of C-OCH$_3$ of anisaldehyde ring | Protons of C-OH Phenolic ring | Aromatic protons | Azomethine protons (-CH=N-) |
|---|---|---|---|---|
| 1 | 3.84 | 8.88 | 6.81-7.17 | 8.62 |
| 1a | 3.73 | - | 6.94-7.78 | 9.79 |
| 1b | 3.80 | - | 7.03-7.74 | 9.73 |
| 1c | 3.68 | - | 7.05-7.96 | 9.81 |
| 1d | 3.77 | - | 6.96-7.87 | 9.86 |
| 1e | 3.76 | - | 7.01-7.83 | 9.79 |

### Antimicrobial activities

Schiff bases, products of the condensation of carbonyl compounds and primary amines have been involved in many biological processes (Singh and Singh 2001). The antimicrobial activity of the Schiff bases and its metal complexes was carried out against two bacteria (*Xanthomoans* sp. and *Staphylococcus aureus VTCC BAA 20*) and two fungal strains (*Fusarium oxysporum* sp. *lycopericireg and Candida albicans*

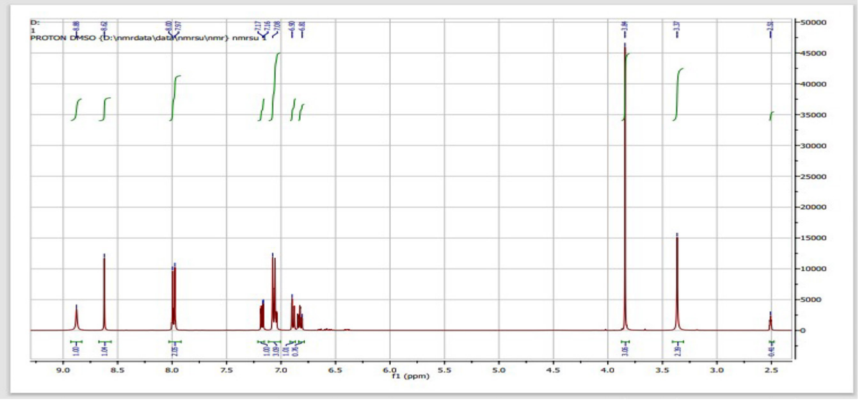


**Fig. 3.** Proton NMR spectrum of Schiff base, ANSAP.

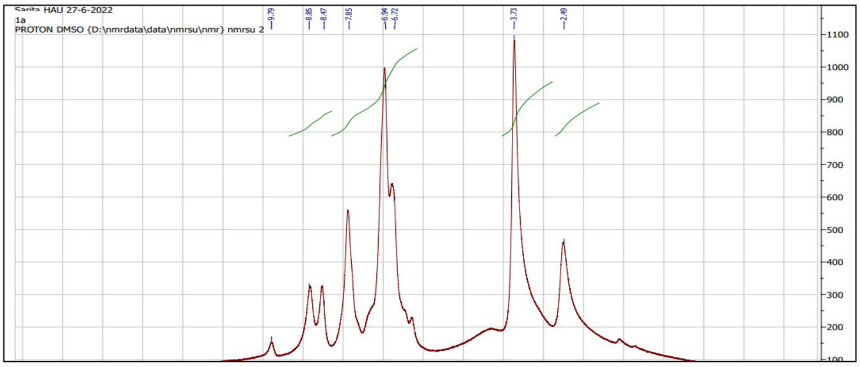


**Fig. 4.** Proton NMR spectrum of complex (1a).

sp.). Zone of inhibition in mm for the Schiff base and its complexes are listed in Table 5 and shown in Fig. 5. From the results of the antibacterial activities, it was found that maximum *Xanthomoans* sp. and *Staphylococcus aureus VTCC BAA 20* growth was inhibited by compounds (1e) which showed inhibition zone of 22 and 18 mm. Maximum *Xanthomoans* sp. growth was also inhibited by compound(1b) and (1d) showing inhibition zone of 21 mm. From the results of the antifungal activities, it was found that maximum *Fusarium oxysporum* sp. *lycopericireg and Candida albicans* sp. growth was inhibited by compound (1c) and (1b) which showed inhibition zone of 30 and 29 mm. (1a) and (1b) also inhibited the greater zone against *Candida albicans* sp. of 28 and 27mm. The free Schiff base was not active against *Fusarium oxysporum* sp. *lycoperici* and showing best results against *Candida albicans* sp. The above results shows that the complexes are more active than free ligand due to chelation effect (Table 5).

## CONCLUSION

Metal complexes of $Mn^{2+}$, $Fe^{2+}$, $Co^{2+}$, $Ni^{2+}$ and $Cu^{2+}$ were synthesized from Schiff base ligand (ANSAP) via condensation reaction. All the synthesized compounds were characterized by elemental analyses, Molar conductance, FT-IR, $^1$H NMR and UV-Visible studies. All complexes were non-hygroscopic in nature. All the compounds were insoluble in water but

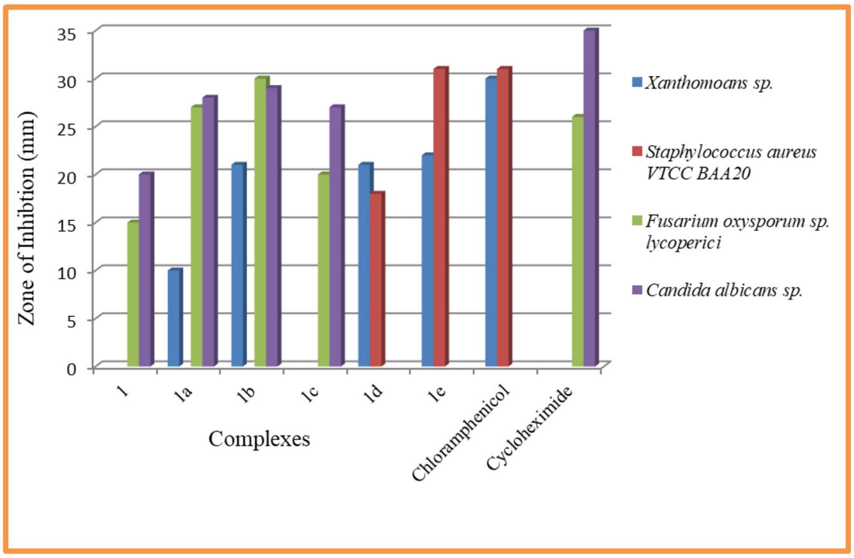


**Fig. 5.** Comparative results of zone of inhibition of ANSAP and its metal complexes.

**Table 5.** Zone of inhibition (mm) of ANSAP and its metal complexes.

| Complex | Zone of inhibition (diameter in mm) | | | |
|---|---|---|---|---|
| | *Xanthomoans* sp. | *Staphylococcus aureus VTCC BAA20* | *Fusarium oxysporum* sp. *lycoperici* | *Candida albicans* sp. |
| 1 | - | - | - | 20 |
| 1a | 10 | - | 15 | 28 |
| 1b | 21 | - | 27 | 29 |
| 1c | - | - | 30 | 27 |
| 1d | 21 | - | 20 | - |
| 1e | 22 | 18 | - | - |
| Chloramphenicol | 30 | 31 | - | - |
| Cycloheximide | - | - | 26 | 35 |

soluble in organic solvents like chloroform, DMSO and DMF, methanol and ethanol. On the basis of all spectral data it was found that ANSAP was neutral bidentate ligand and all the complexes showed tetrahedral geometry. The antimicrobial activity of the ligand and their metal complexes were screened out against bacterial and fungal strains. The study shows that metal complexes showed more inhibition activity as compared to ligand.

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