



SYNTHESIS, CHARACTERIZATION, ELECTRICAL CONDUCTIVITY, BIOLOGICAL AND CATALYTIC ACTIVITY OF SOME COORDINATION POLYMERS OF SALEN TYPE SCHIFF BASE

Ashish D. Bansod

Department of Chemistry,

Rajarshee Shahu Science College Chandur Rly 444904, India

ABSTRACT

The Schiff bases ligand 4,4'-bis-[(N-O-tolulylsalicylaldiamine-5)azo]biphenyl (H_2BTSAB) and its chelate polymers with Cr(III), Mn(III), Fe(III), VO(IV), Zr(IV), Th(IV), Ti(III) and $UO_2(V)$ have been prepared. These have been characterized by elemental analyses, IR and electronic spectra, magnetic susceptibility measurements and thermogravimetric analysis. Thermogravimetric analysis confirms the coordination of H_2O in complexes. 1H NMR spectrum of ligand clearly indicates presence of OH and azomethine groups. The ligand act as a bidentate molecules coordinating through phenolic oxygen thermal data have been analysed for the kinetic parameters by using Broido method and activation energy has in the range 33.18 to 80.42 $kJmol^{-1}$ while the decomposition follows first order kinetics. The 40-220 $^{\circ}C$ and the chelates were found to show semiconducting behavior. The activation energy of the electrical conduction lies in the range 1.233-0.127eV. Oxidation of styrene with selected catalysts was tested using H_2O_2 as an oxidant.

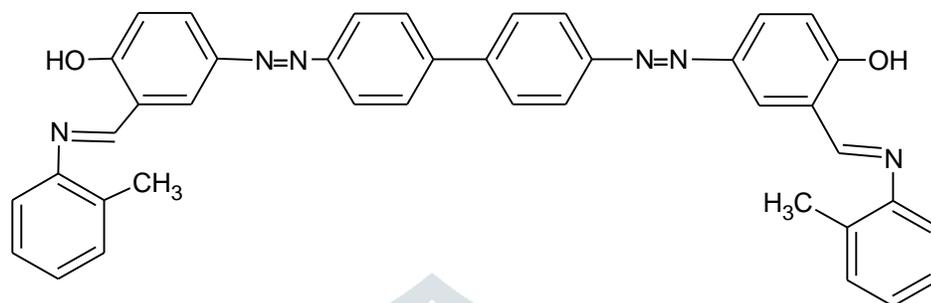
Keywords: Schiff base, polychelates, TGA, solid-state conductivity, catalytic activity.

INTRODUCTION

Schiff bases on the divalent metal Chelates with symmetric as well as unsymmetrical Schiff base ligands have been reported extensively due to their biological relevance¹⁻⁴. Even metal complexes of the ligands having azo groups find a place in the literature due to their use as model biological systems. But the literature on the metal with Schiff base ligands having azo group is limited⁵⁻⁶. The synthesis and characterization of divalent metal chelates are quite common however; complexes of higher valent metal ions are limited to a few reports⁷⁻⁸.

The growing interest on polychelates inspired us to use the bis-bidentate ligand (1) to prepare polychelates of various geometry with tri- tetra and hexavalent metal ions in order to explore the effect of electron donating

methyl groups on the chelating behavior and to investigate their biological activity. In the present paper we describe the synthesis of Cr(III), Mn(III), Fe(III), VO(IV) Zr(IV), Th(IV), Ti(III) and UO₂(V) complexes with ligand complexes H₂BTSABZ(1) and their characterized by elemental analyses, IR and electronic spectra, magnetic susceptibility measurements and thermogravimetric analysis.



Scheme (I)

1 EXPERIMENTAL

1.1 Reagents and materials

All Chemicals used were of AR grade and the solvents were used after distillation. Microanalyses of C, H and N were carried out at the Regional Sophisticated Instrumentation Centre (RSIC), Punjab University, Chandigarh, India. The metal contents were determined by standard method⁹⁻¹⁰, after decomposing the chelates with conc. HNO₃.

1.2 Physical measurements

¹H NMR spectra of the ligand was taken using TMS as the internal standard on a 90 MHz Perkin–Elmer R-32 spectrometer. The IR 400–4000 cm⁻¹ at RSIC, Punjab University, Chandigarh, India, magnetic measurements were carried out at room temperature by Gouy method using Hg[Co(SCN)₄], as celebrant. The reflectance spectra of the solid compounds suitably diluted with MgO, were recorded on a Carl-Xeiss DMR-21 spectrometer at RSIC Indian Institute of technology Chennai, India. Thermogravimetric analyses of the complexes were carried out on a simple manually operated thermo balance fabricated in our laboratory. The instrument was calibrated using crystalline copper sulphate pentahydrate. Samples were run in air atmosphere with a heating rate of 10⁰C min⁻¹. The electrical conductivity was measured in pellets using a dc micro voltmeter¹¹⁻¹².

Catalytic Study: Epoxidation of styrene

Catalytic oxidation of styrene to corresponding epoxide by Mn(III) and Fe(III), polychelates were studied in presence of an aqueous solution of 30% H₂O₂ as an oxidant. All catalytic reactions were carried out in 50 mL reaction flask fitted with water condenser. A general procedure was followed for all reactions. In a typical experiment, styrene (5.2 g, 0.05 mol) were mixed in 5 mL of acetonitrile and the reaction mixture was heated at 40⁰C with continuous stirring in a water bath. An appropriate catalyst to be tested (0.025 g) was added to the reaction mixture and heated at 40⁰C under constant stirring for 24 h. After the completion of the reaction an organic layer was separated by centrifugation and analyzed using a Shimadzu 14B Gas Chromatography with SE-30 column and FID detector.

1.3 Synthesis of 4, 4'-bis-[(N-O-tolulylsalicylaldiamine-5)azo]biphenyl

The ligand was prepared by the method reported in literature with slight modification the dye 4,4'-bis-[(bis(salicylaldehyde-5)azo) biphenyl was prepared by diazotization[13].The dye (2.5g, 0.005mol) was dissolved in 25ml methanol .A solution of o-toluidine (1:1g,0.01mol) in 25ml ethanol was added to it (mole ratio 1:2) with constant stirring. A drop of Conc. H₂SO₄ was added as a catalyst and the reaction mixture was refluxed in a water bath for about 3hr. The Schiff base ligand 4,4'-bis-[(N-O-tolulylsalicylaldiamine-5)azo]biphenyl (H₂BTSAB) was obtained as greenish brown product. The purity of the compounds was checked by thin layer chromatography (TLC). It was characterized by elemental analysis, IR, UV, ¹H NMR and mass spectra. It was crystallized from 1:1 DMF ethanol mixture, yield 2.5g (80), m.p.235⁰C.

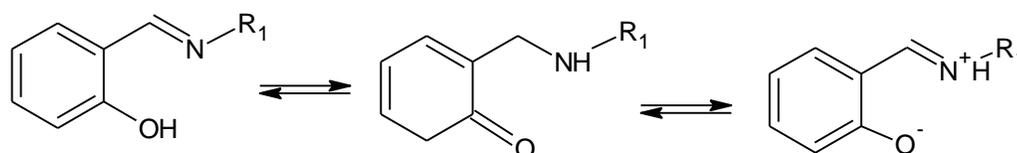
2.4 Synthesis of Polychelates

The polychelates were prepared reaction of the metal salts CrCl₃.6H₂O, Mn(OAc).2H₂O, FeCl₃, VOSO₄.5H₂O, ZrOCl₂.8H₂O, Th(NO₃)₄.5H₂O, TiCl₃ and UO₂(NO₃)₂.6H₂O. with Schiff base ligand. To a solution of the ligand (3.2g, 0.005mol) in DMF (25ml) a solution of the metal salts (0.005mol) in ethanol (25ml) was added within DMF (25ml) a solution of the metal (0.005mol) in ethanol (25ml) was added with constant stirring. The mixture was refluxed on an oil bath for 2-3h .The resulting polychelates were filtered, washed several times with hot water, hot DMF, ethanol and finally with acetone to remove unreacted reactants and dried at 100⁰C in an electric oven for 45 minutes, yields 60-65⁰C (Table1).

2 RESULTS AND DISCUSSION

The reaction of 4, 4'-bis-[(salicylaldehyde-5)azo]biphenyl with o-toluidine in DMF ethanol mixture yields the Schiff base (H₂BTSAB) (1), the formulation of which is supported by analytical and spectral data. Its ¹H NMR spectrum in DMSO-d₆ shows signals at 7.16, 8.10, 8.25 and 8.74ppm (δ) corresponding to phenyl -C=N and phenolic protons, respectively¹³⁻¹⁴. The signal due to CH, protons observed at 1.27ppm (δ). The IR spectrum of the Schiff bases shows bands at 1616, 1483, 1282 and 3030cm⁻¹ corresponding to ν(C=N), ν(N=), ν(C-O) and ν (O-H) frequencies, respectively¹⁵.

The ligand H₂BTSAB reacts with metal salts 1:1 mol ratio resulting in the formation of polychelates according to the general equation (1).The elemental analyses (table1) indicate 1:1 (metal: Ligand) stoichiometry. The resulting polychelates are coloured solids and insoluble in water and most of the organic solvents, but are sparingly soluble in DMF and DMSO only.



(H₂BTSAB)

Scheme (III)

For M= Cr(III),Fe(III),Zr(IV),Th(IV),Ti(III) : X=Y=H₂O

VO(IV) ; X= H₂O Y=O

Mn(III) X =OAc and Y=Nil

UO₂(VI) :X=Y=O (H₂BTSAB)

2.1 IR spectra

The ligand (H₂BTSAB) shows a broad intense band at 3030cm⁻¹ assignable to intermolecular, H-bonded phenolic OH stretching vibrations, which are absent in the spectra of the polychelates, indicating the coordination of the ligand with the metal ion through the phenolic oxygen atom¹⁶. This is further supported by the shift to the higher frequency (26-38cm⁻¹) of ν (C-O) vibrations from 1282cm⁻¹ in ligand to 1308-1320cm⁻¹ in the polychelates, and also confirmed by the appearance of a new, low intensity band at 517-558cm⁻¹ in the spectra of the polychelates due to, there by indicating ν (M-O) vibrations. A strong and sharp band at 1616cm⁻¹ in the spectrum of the ligand due to ν (C=N), shows a shift a lower frequency by due to the ligand due to 20cm⁻¹ in the spectra of the polychelates, thereby indicating the coordination to the metal ions by the azomethine nitrogen¹⁷⁻¹⁸. This is again supported by the appearance of a new, low intensity band at 400-466 cm⁻¹ due to ν (M-N) vibrations owing to the formation of a new metal ligand bond. Strong and sharp bands at 912 and 905 cm⁻¹ in the spectra of the oxovanadium and dioxouranium polychelates, may be attributed to ν (V=O) and ν_{as} (V=O) modes respectively. A moderately sharp band at 1483cm⁻¹ in the spectra of the ligand and the polychelates due to ν (N=N) and no change in its position shows the indifferent nature of the azo group towards coordination.

A broad and strong band in the polychelates of Cr(III), Fe(III), VO(IV), Zr(IV), Th(IV) and Ti(III) in the region 3367-3400cm⁻¹ due to ν (O-H) and sharp shoulder at 1650-1656cm⁻¹ due to ν (H₂O) indicate the presence of water molecules and a strong sharp band at 830cm⁻¹ due to the ν (H₂O) racking mode suggests these water molecules as coordinated ones. Two additional bands are observed at 1522 and 1391cm⁻¹ in Mn(III) polychelates which may be assigned to ν (OCO) and ν_{as} (OCO), which is considered a diagnostic band for a monodentate ligand. Thus, the IR data suggest a dibasic bis bidentate nature of the ligand and its coordination to metal ions through the phenolic oxygen and azomethine nitrogen atoms.

2.2 Thermogravimetric analysis

Thermal decomposition studies of polychelates have been carried out to corroborate the information obtained from IR spectra; studies about the status of water molecules present in some these polychelates as well as know their decompositions pattern. In the case Cr(III),Fe(III),Zr(IV),Th(III) and Ti(III) polychelates, elimination of two water molecules takes place between 140-330⁰C indicating the presence of two coordinated water molecules, while the VO(VI) polychelates loses one coordinated water molecules in this stage. The polychelates of Mn(III) and UO₂(VI) show negligible weight up to 300⁰C indicating the absence of any water molecules. The second stage of the decompositions is fast and proceeds at 400-600⁰C leading to the formation of the respectively metal oxides. The thermal stability order of the polychelates, on the basis of the procedural decomposition temperature is Ti>Cr>UO₂>Mn=Zr>VO>Th>Fe (Table 1).

The order thermal decomposition and corresponding activation energies have been evaluated from the figures

generated using Broido method¹⁹. The plots of $\ln[\ln(1/y)]$ vs $1/T$ are linear corresponding to first order kinetics where the fraction not yet decomposed (i.e. residual weight fraction), $y=(w_t-w_\infty) / (w_0-w_\infty)$, where w_t is the weight of the substance at temperature $t^\circ\text{C}$, w_0 is the weight of the substance at initial state and w_∞ is the weight the residue at the end of decomposition. The slopes of the plots yield the thermal activation energy (E_a) of decomposition, which lies between 33.18 and 80.42 kJmol^{-1} .

2.3. Electronic spectra and magnetic moment

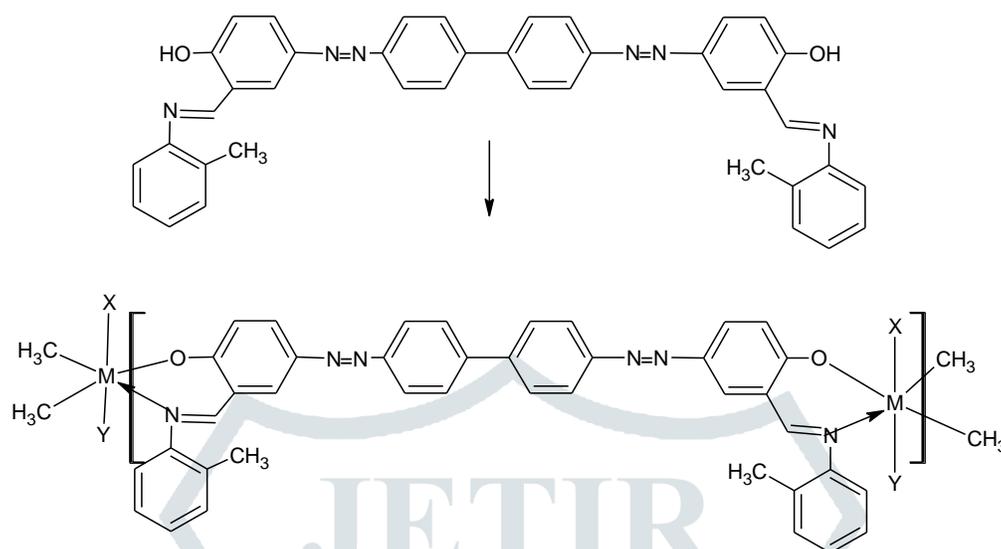
The magnetic moments of the polychelates are given Table 1. The observed value been evaluated from for the Cr(III) polychelates is 4.01 B.M. in accordance with spin-only value for three unpaired electrons corresponding to high-spin octahedral Cr(III) complexes²⁰. The Mn(III) polychelates has a magnetic moment of 4.94 B.M., indicating the presence of four unpaired electrons and that of Fe(III) 5.90 B.M., indicating the high-spin state of Fe(III). The magnetic moments of 1.73 and 1.80 B.M. of VO(IV) and Ti(III) polychelates, respectively suggest the presence of one unpaired electrons, the higher value may be due to orbital contributions.

The diffuse reflectance spectrum of the Cr(III) polychelates exhibits three bands at 16,720, 22,420 and 28,090 cm^{-1} which may be assigned to ${}^4A_{2g} \rightarrow {}^4T_{2g}$ (F), ${}^4A_{2g} \rightarrow {}^4T_{1g}$ (F) and ${}^4A_{2g} \rightarrow {}^4T_{2g}$ transitions, respectively, for an octahedral stereochemistry²¹. The Mn(III) polychelates shows an intense band at 27,780 cm^{-1} which may be due to a ligand to metal charge transfer transition. In the second region three moderate to weak d-d bands are observed 13,420, 16,390 and 18,180 cm^{-1} assigned ${}^5B_1 \rightarrow {}^5B_2$, ${}^5B_1 \rightarrow {}^5A_1$ and ${}^5B_1 \rightarrow {}^5E$ transitions respectively, suggesting square-pyramidal geometry around the Mn(III) ion. The electronic spectrum of Fe(III) polychelates displays three bands at 11,300, 21,505 and 27,030 cm^{-1} assignable to ${}^6A_{1g} \rightarrow {}^6A_{1g}$, ${}^6A_{1g} \rightarrow {}^4T_{2g}$ and ${}^6A_{1g} \rightarrow {}^4E_g$ transitions, respectively indicating that the polychelates processes a high-spin octahedral configuration²². Three d-d transition bands are observed in the polychelates of VO(IV) which may be assigned as 10,200 cm^{-1} $d_{xy}(b_2) \rightarrow d_{yz}$, $d_{yz}(e^0)$, 18,870 cm^{-1} $d_{xy}(b_2) \rightarrow d^2_{x^2-y^2}(b_1^*)$ and 26,450 cm^{-1} $d_{xy}(b_2) \rightarrow d_{z^2}(a_1^*)$ towards octahedral geometry²³. Only one band has been observed at 18,660 cm^{-1} derived from the transition ${}^2T_2 \rightarrow {}^2E_g$ for an octahedral symmetry in the Ti(III) polychelates. The broad and double hump nature of the spectrum indicates the presence of Jahn Teller distortion. The Zr(IV), Th(IV) and UO₂(VI) polychelates, as expected are diamagnetic. Their electronic spectra do not exhibit any characteristic d-d transition except charge transfer bands.

2.4. Solid electrical conductance

The measurement of the solid electrical conductance of Schiff base ligand and its polychelates was carried out in their pellet form from room temperature to about 493 K. The results of the electrical conductivity and activation energy are incorporated in Table 2. The room temperature electrical lies in the range 3.82×10^{-11} to $1.05 \times 10^{-13} \Omega^{-1}\text{cm}^{-1}$ indicating their semiconducting nature²⁴ and decreases in the order $\text{Zr} > \text{VO} > \text{Cr} > \text{Th} > \text{BTSAB} > \text{Ti} > \text{Mn} > \text{Fe} > \text{UO}_2$. The electrical conductivity (σ) varies exponentially with the absolute temperature according to the relationship $\sigma = \sigma_0 \exp(-E_a/kT)$ where σ_0 is a constant, E_a activation energy of conduction process. T the absolute temperature and k the Boltzmann constant. When $\log \sigma$ is plotted against $1/T$ a linear dependence was observed, also suggesting the semiconducting behavior of the ligand and its polychelates. Here, the lower temperature region has lower slopes with lower slopes

with lower activation energies and the higher temperature region has higher slopes with higher E_a values. This break observed in plots. i.e. phase transition, in case of the ligand, may be the ligand, may be attributed to two molecular structures having keto and enol forms which exchange the conduction at different thermal stabilities [Eq(2)]



Scheme (III)

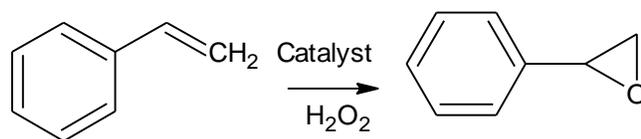
In the case of the polychelates the lower values of $\log \sigma$ in the low temperature region may be attributed to extrinsic conduction present in them, where the conduction is due to excitation of carries from the donor localized level to the conduction band. Whereas at higher temperature these may behave as intrinsic semiconductors in which the carries are thermally activated from the valence band to the conduction band. This may be because of the interaction between the electrons of d orbital of the metal and π orbital of the ligand higher temperature, which leads to localized action of the π electronic charge on ligand that tends to increase the activation energy²⁵⁻²⁷. The activation energy of electrical conduction is calculated from the slopes of these plots in the high temperature region, which lies in the range between 1.233 and 0.127eV.

2.5 Biological Activity

Antimicrobial activities of the ligands and the complexes have been carried out, against the bacteria *E. coli*, *Bacillus sp.*, *Staphylococcus* and *Pseudomonas sp.* using nutrient agar medium by the disc diffusion method. The test solution were prepared in DMSO to a final concentration of 0.01% and 0.02% and soaked in filter paper of 5mm diameter and 1 mm thickness. These discs were placed on the already seeded plates and incubated at 35°C for 24 hours²⁸. The diameter (mm) of the inhibition zone around each disc was measured after 24 hours and results are listed (Table-3 and Fig.5) Streptomycin was used as standard. It was observed from these studies that the metal complexes have a higher activity than the free ligands against the same microorganism under the identical experimental conditions. The mode of action of the complexes may involve the formation of hydrogen bonds involving the azomethine group with microbial or ribosomes of microbial cells resulting in interference with normal cell processes. The toxicity increase with increasing concentration of the complexes²⁹⁻³⁰.

2.6 Catalytic activity

The catalytic activity of Mn(III), Fe(III,) complexes towards the epoxidation of styrene using H₂O₂ as an oxidant resulted in only 12-15 % conversion of styrene into styrene oxide(III). Optimization of the reaction conditions by changing amount of substrate, oxidant and catalyst as well as temperature of the reaction did not much improve the yield of the styrene oxide formation³¹⁻³⁶.



Oxidation product of styrene (III)

CONCLUSION

The formation of macromolecular chain may be expected for a ligand with two chelating sites, which for steric reasons cannot interact with the same metal ion. The formation of chelate polymer is also supported by the fact that all chelates obtained are insoluble in water and common organic solvents. The extended nature of TG curves showing a slow decomposition in wide range of temperature also indicating their polymeric nature.

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Table 1: Analytical and physical data of the polychelates

| Compound | Formula | μ_{eff} | Decomposition Temp($^{\circ}\text{C}$) | Ea (kJMol $^{-1}$) | Yield (%) | Elemental Analysis | | | |
|---|---------|--------------------|---|---------------------|-----------|--------------------|----------------|------------------|------------------|
| | | | | | | C | H | N | M |
| H ₂ BTSAB (Green Brown) | 628.00 | --- | 360 | 33.18 | 80 | 76.00 (76.43) | 5.00 (5.10) | 13.00 (13.38) | -- |
| [Cr(H ₂ BTSAB)(H ₂ O)]Cl (Light Brown) | 713.99 | 4.01 | 340 | 52.04 | 65 | 66.00 (66.47) | 4.25 (4.37) | 12.00 (12.24) | 7.02 (7.28) |
| [Mn(H ₂ BTSAB)(OAC)] (Light Brown) | 716.94 | 4.94 | 310 | 80.36 | 60 | 67.67 (68.11) | 4.34 (4.46) | 12.00 (11.35) | 7.11 (7.42) |
| [Fe(H ₂ BTSAB)(H ₂ O)]Cl (Light Brown) | 717.85 | 5.90 | 270 | 80.42 | 65 | 67.57 (68.59) | 4.53 (4.60) | 11.53 (11.67) | 7.49 (7.60) |
| [VO(H ₂ BTSAB)(H ₂ O)] (Light Brown) | 692.94 | 1.73 | 290 | 58.85 | 60 | 67.25 (67.35) | 4.25 (4.30) | 11.28 (11.35) | 6.91 (6.95) |
| [Zr(H ₂ BTSAB)(H ₂ O)] (Yellowish Brown) | 753.22 | Dia | 310 | 34.96 | 62 | 63.59 (63.65) | 4.34 (4.40) | 10.80 (10.95) | 11.79 (11.85) |
| [Th(H ₂ BTSAB)(H ₂ O)](NO ₃) ₂ (Golden Brown) | 894.04 | Dia | 280 | 40.35 | 63 | 53.33 (53.40) | 3.49 (3.50) | 9.03 (9.10) | 25.63 (25.70) |
| [Ti(H ₂ BTSAB)(H ₂ O)]Cl (Yellowish Brown) | 709.88 | 1.81 | 390 | 46.76 | 60 | 67.22 (67.25) | 4.37 (4.40) | 11.69 (11.75) | 6.53 (6.70) |
| [UO ₂ (H ₂ BTSAB)] (Maroon) | 896.03 | Dia | 320 | 39.75 | 60 | 53.17 (53.20) | 3.04 (3.09) | 8.73 (8.75) | 26.26 (26.58) |

Table 2: Electrical conductivity data of Schiff base and its polychelates

| Compound | Electrical Conductivity ($\Omega^{-1}\text{cm}^{-1}$) | Temperature (K) | Activation Energy (eV) |
|---|---|-----------------|------------------------|
| H ₂ BTSAB | 5.42×10^{-12} | 40 | 0.512 |
| | 3.47×10^{-10} | 220 | |
| [Cr(H ₂ BTSAB)(H ₂ O)]Cl | 1.69×10^{-11} | 40 | 0.044 |
| | 1.85×10^{-9} | 220 | |
| [Mn(H ₂ BTSAB)(OAC)] | 3.47×10^{-12} | 40 | 0.902 |
| | | 220 | |
| [Fe(H ₂ BTSAB)(H ₂ O)]Cl | 1.27×10^{-12} | 40 | 0.687 |
| | 2.85×10^{-11} | 220 | |
| [VO(H ₂ BTSAB)(H ₂ O)] | 3.15×10^{-11} | 40 | 1.233 |
| | 8.80×10^{-11} | 220 | |
| [Zr(H ₂ BTSAB)(H ₂ O)] | 3.82×10^{-12} | 40 | 12.33 |
| | 8.80×10^{-11} | 220 | |
| [Th(H ₂ BTSAB)(H ₂ O)](NO ₃) ₂ | 1.05×10^{-12} | 40 | 0.990 |
| | 1.96×10^{-11} | 220 | |
| [Ti(H ₂ BTSAB)(H ₂ O)]Cl | 3.52×10^{-12} | 40 | 0.349 |
| | 2.40×10^{-10} | 220 | |
| [UO ₂ (H ₂ BTSAB)] | 1.05×10^{-12} | 40 | 0.219 |
| | 1.96×10^{-11} | 220 | |

Table 3: Antimicrobial data of ligand and their chelates

| Compound | <i>E coli</i> (mm) | <i>Bacillus sp</i> (mm) | <i>Pseudomonous</i> sp.(mm) | <i>Staphylocous</i> (mm) |
|------------------|-----------------------|----------------------------|--------------------------------|-----------------------------|
| H ₂ L | 6.8 | 5.6 | 6.7 | 6.9 |
| CrL | 9.12 | 8.10 | 9.11 | 9.10 |
| MnL | 10.11 | 10.13 | 12.13 | 10.12 |
| FeL | 9.11 | 8.12 | 9.12 | 11.12 |
| Streptomycin | 18.21 | 20.23 | 18.21 | 18.22 |

Fig.5: Biological studies of ligand and their Chelates

