



## Synthesis, spectral, thermal and antimicrobial studies of some transition metal complexes containing 2-thiophenecarboxaldehyde moiety

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### ABSTRACT

Some Schiff base metal complexes of Co(II), Ni(II) and Cu(II) derived from 2-thiophenecarboxaldehyde with 2-imidodicarbonic diamide (TID) 2-chloro-4-nitroaniline (TCA) have been synthesized and characterized by elemental analysis, FT-IR, molar conductance, electronic spectra, ESR, magnetic susceptibility, and thermal analysis. The complexes exhibit coordination number 4 or 6. The complexes are coloured and stable in air. Analytical data revealed that all the complexes exhibited 1:1 or 1:2 (metal: ligand) ratio. Thermal data show degradation pattern of the complexes. The Schiff base and metal complexes show a good activity against the Gram-positive bacteria; *Staphylococcus aureus* and Gram-negative bacteria; *Escherichia coli* and fungi *Aspergillus niger* and *Candida albicans*. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the Schiff bases.

**Keywords:** Synthesis; 2-Thiophenecarboxaldehyde; Thermal analyses; Biological activity

### INTRODUCTION

A large number of Schiff bases and their metal complexes have been found to possess important biological and catalytic activity. Due to their great flexibility and diverse structural aspects, a wide range of Schiff bases have been synthesized and their complexation behavior was studied. Metal complexes of Schiff bases containing nitrogen and sulphur as donor ligand atoms are of interest in different fields like industry, pharmacy and biology. It is well known that N and S play a key role in the coordination of metals at the active sites of numerous biomolecules. They are used as models for biological systems and find applications in biomimetic catalytic reactions. Heterocyclic compounds are very widely distributed in nature and are essential to many fields of material science and biological systems (Chohan, 1999; Garg, 2006; Bagihalli, 2009). 2-thiophenecarboxaldehyde are a remarkably versatile group of compounds that have many biological activities and found recent applications in the drug development. On the industrial scale, they have wide range of applications such as dyes and pigment. Metal complexes with these ligands are becoming increasingly important as biochemical, analytical and antimicrobial reagents in the design of molecular models, material chemistry and so on (Coombes, 2005; Mahajan, 2009;

Chohan, 2001).

### Experimental

**Materials and instrumentation:** All the used chemicals and solvents were of Anal R grade. All the reagents used for the preparation of the Schiff bases were obtained from Sigma Aldrich. Metal salts were purchased from Loba Chemie. Elemental analyses were performed on an Elementar Vario EL III Carlo Erba 1108 analyzer. Electronic spectra (in DMSO) were recorded on Perkin Elmer Lambda-2B-spectrophotometer. Molar conductance measurements were conducted using  $10^{-3}$  M solution of the complexes in DMSO on Elico-CM 82 Conductivity Bridge at room temperature. Magnetic susceptibility measurements were carried out on a Gouy balance at room temperature using mercuric tetrathiocyanatocobaltate(II) as the calibrant. Diamagnetic corrections were applied in compliance with Pascal's constant. FT-IR spectra were recorded in KBr medium on a Perkin Elmer RX1 spectrophotometer in wave number region 4000-400  $\text{cm}^{-1}$  X-band EPR spectra were recorded on a Varian E-112 spectrometer at room temperature operating at the X-band region with 100 kHz modulation frequency, 5 mw microwave power and 1 G modulation amplitude using TCNE as the internal standard. Thermogravimetric analysis was carried out under atmospheric condition with a heating rate  $10^\circ\text{C min}^{-1}$  on TGA Q500 universal V4.5A TA instrument.

**Synthesis of Schiff bases:** The Schiff bases (TID and TCA) have been synthesized by adding the ethanolic solution of 2-thiophenecarboxaldehyde with 2-Imidodicarbonic diamide (TID) 2-chloro-4-nitroaniline (TCA) in equimolar ratio. The reaction mixture was

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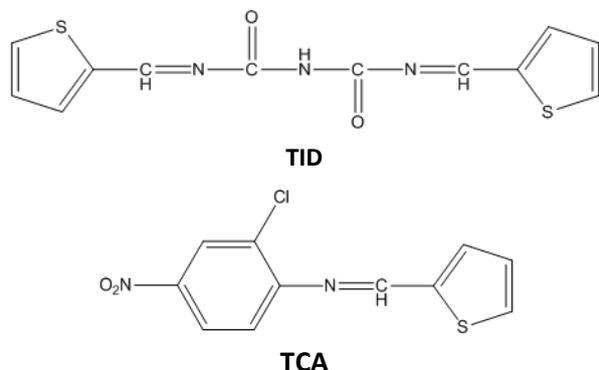
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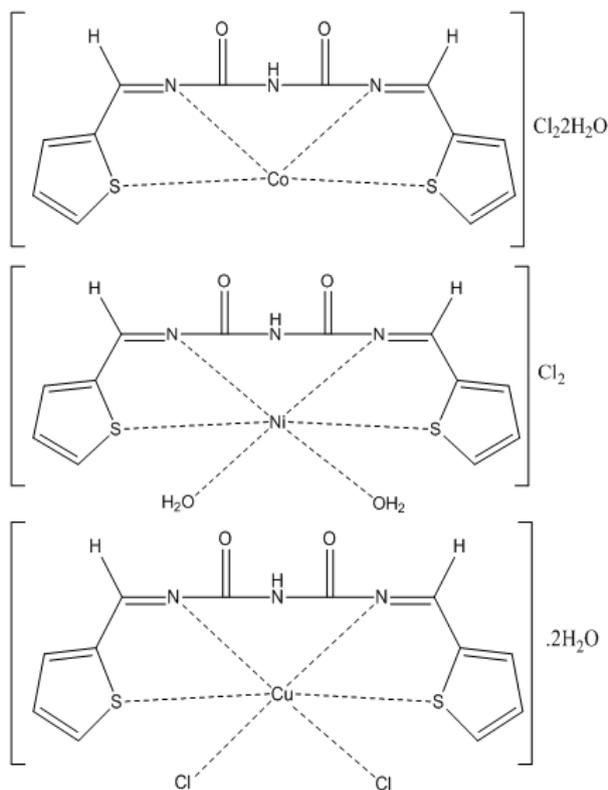
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then refluxed on water bath for about 3–4 hours. The condensation product was filtered, thoroughly washed with ethanol and ether, recrystallized and dried in vacuo. The Purity of the synthesized compound was monitored by TLC using silica gel G (Yield: TID = 80%; TCA = 83%).

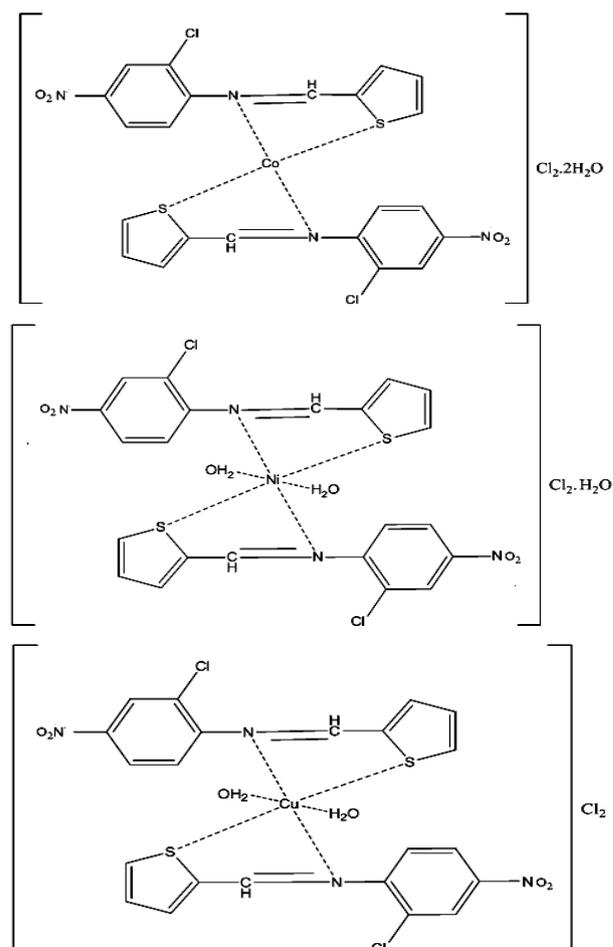


**Figure 1: Suggested structure of Schiff base legends**

**Synthesis of metal complexes:** The metal complexes were prepared by the mixing of (50 ml) ethanolic solution of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ / $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ / $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  with the (50 ml) ethanolic solution of Schiff bases (TID/TCA) in 1:1 or 1:2 (metal:ligand) ratio. The resulting mixture was refluxed on water bath for 6-9 h. A coloured product appeared on standing and cooling the above solution. The precipitated complex was, filtered washed with ether and recrystallized with ethanol several times and dried under the reduced pressure over anhydrous  $\text{CaCl}_2$  in a desiccator. It was further dried in electric oven at 50-70°C (yield: 59.6-69.0%).



**Figure 2: Proposed structure metal complexes of TID**



**Figure 3: Proposed structure metal complexes of TCA**

**Biological activity:** The *in vitro* biological activity of the investigated Schiff base and its metal complexes was tested against the bacteria *Escherichia coli* and *Staphylococcus aureus* by disc diffusion method using nutrient agar as medium and streptomycin as control. The antifungal activities of the compounds were also tested by the Well diffusion method against the fungi *Aspergillus niger* and *Candida albicans*, on potato dextrose agar as the medium and miconazole as control. The stock solution was prepared by dissolving the compounds in DMSO. In a typical procedure, a well was made on agar medium inoculated with microorganism. The well was filled with the test solution using a micropipette and the plate was incubated 24 h for bacteria at 37 °C and 72 h for fungi at 30°C. During this period, the test solution diffused and the growth of the inoculated microorganism was affected. The inhibition zone was developed, at which the concentration was noted.

## RESULTS AND DISCUSSION

All the metal complexes are coloured, solid and stable towards air and moisture at room temperature. They decompose on heating at high temperature and more or less soluble in common organic solvents. Analytical and physical data of the compounds are given in the Table 1. All the metal chelates have 1:1 or 1:2 (metal:ligand) stoichiometry.

Table 1: Analytical and physical data of the compounds

Molcular Formula/ Mol. Wt./ ( Colour)	Yield (%)	Elemental Analysis; Found/ Calculated (%)				* $\Lambda_m$
		C	H	N	M	
C <sub>12</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub> [TID] 291 (Light yellow)	80	49.91 (49.48)	3.08 (3.09)	14.38 (14.43)	-	-
[Co(C <sub>12</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub> )]Cl <sub>2</sub> .2H <sub>2</sub> O 458 (Indigo)	68	31.37 (31.5)	2.83 (2.84)	9.13 (9.19)	12.83 (12.89)	121.6
[Ni(C <sub>12</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub> )(H <sub>2</sub> O) <sub>2</sub> ].Cl <sub>2</sub> 457 (Light Green)	59	31.37 (31.5)	2.83 (2.84)	9.15 (9.19)	12.75 (12.80)	136.2
[Cu(C <sub>12</sub> H <sub>9</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub> )Cl <sub>2</sub> ].2H <sub>2</sub> O 461 (Dark Green)	65	30.9 (31.23)	2.78 (2.81)	9.01 (9.11)	13.63 (13.78)	51.5
C <sub>11</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ClS [TCA] 267 (Yellow)	83	49.07 (49.43)	2.60 (2.62)	10.43 (10.48)	-	-
[Co(C <sub>11</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ClS) <sub>2</sub> ].Cl <sub>2</sub> .2H <sub>2</sub> O 709 (Yellow)	65	37.66 (37.76)	2.56 (2.57)	7.98 (7.01)	8.40 (8.20)	121.2
[(Ni(C <sub>11</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ClS) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]. Cl <sub>2</sub> .H <sub>2</sub> O 719 (Green)	63	36.71 (36.92)	2.78 (2.72)	7.78 (7.83)	7.58 (7.60)	116.4
[Cu(C <sub>11</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> ClS)(H <sub>2</sub> O) <sub>2</sub> ].Cl <sub>2</sub> 705 (Yellow)	68	37.44 (37.23)	2.55 (2.53)	7.94 (7.89)	8.60 (8.96)	128.3

$$*\Lambda_m = (\Omega^{-1} \text{cm}^2 \text{mol}^{-1})$$

Table 2: Antibacterial screening data for the legends and their complexes

Compound	Diameter of inhibition zone (mm), Concentration in ppm											
	<i>E. coli</i>						<i>S. aureus</i>					
				% Activity index*						% Activity index*		
	25	50	100	25	50	100	25	50	100	25	50	100
TID	11	14	17	50	58	61	10	13	15	56	59	63
Co(II)	16	18	22	73	75	79	14	17	19	78	77	79
Ni(II)	15	17	21	68	71	75	12	16	18	67	73	75
Cu(II)	17	20	24	77	83	86	14	18	20	78	82	83
TCA	12	14	16	55	58	57	12	15	18	67	68	75
Co(II)	17	20	25	77	83	89	15	19	21	83	86	88
Ni(II)	14	16	20	64	67	71	13	17	19	72	77	79
Cu(II)	16	19	23	73	79	82	14	18	20	78	82	83
Streptomycin (Standard)	22	24	28	100	100	100	18	22	24	100	100	100

$$*\% \text{ Activity Index} = \frac{\text{Zone of inhibition by test compound (diameter)}}{\text{Zone of inhibition by standard (diameter)}} \times 100$$

**IR spectra:** The IR spectra of the complexes were compared with those of the free ligand in order to determine the involvement of coordination sites in chelation. Characteristic peaks in the spectra of the ligand and complexes were considered and compared.

Spectrum of the TID ligand (Schiff base) for IR spectra shows a medium band at 1614 cm<sup>-1</sup> due to  $\nu(\text{C}=\text{N})$  azomethine group which has shifted to lower frequency by 16-24 cm<sup>-1</sup> in the complexes indicating its participation in chelation through azomethine nitrogen. The lowering of band is due to the reduction of electron density in the azomethine link. Thiophene ring  $\nu(\text{C}-\text{S}-\text{C})$  shows absorption band at 852 cm<sup>-1</sup> in the Schiff base spectrum. These bands show lower shift by 16-25 cm<sup>-1</sup> in all complexes, suggesting participation of thiophene  $\nu(\text{C}-\text{S}-\text{C})$  sulphur in complexation. The IR spectra of Schiff base ligand exhibit a strong intensity band at

1683 cm<sup>-1</sup> due to (C=O) amide. These bands are unchanged after complexation. This indicate that non involvement of (C=O) amide group in complexation The IR spectra of complex exhibit new band around 3412 cm<sup>-1</sup>-3369 cm<sup>-1</sup> which may be due to  $\nu_{\text{stre}}$  water molecules. A band of medium intensity at 812 cm<sup>-1</sup>  $\nu_{\text{OH}}$  rocking suggests the presence of coordinated water in nickel(II) complex. These bands are absent in the cobalt and copper complexes. The new bands in all complexes at 498±2 and 410±4 cm<sup>-1</sup> have been assigned to  $\nu(\text{M}-\text{N})$  and  $\nu(\text{M}-\text{S})$  modes respectively (Dubey, 2008; Raman, 2007; Ourari, 2006; Garg, 2003; Mahajan, 2007)

IR spectrum of TCA ligand shows a band at 1624 cm<sup>-1</sup> due to  $\nu(\text{C}=\text{N})$  azomethine group, which shifts down by 22-34 cm<sup>-1</sup> in all complexes. This suggests coordination through azomethine nitrogen. Thiophene ring  $\nu(\text{C}-\text{S}-\text{C})$  shows absorption band at 850 cm<sup>-1</sup> in the Schiff base

**Table 3: Antifungal screening data for the legends and their complexes**

Compound	Diameter of inhibition zone(mm), Concentration in ppm					
	<i>A. niger</i>			<i>C. albicans</i>		
	25	50	100	25	50	100
TID	12	15	19	11	14	17
Co(II)	16	22	27	16	18	23
Ni(II)	15	20	24	15	17	22
Cu(II)	15	21	25	18	20	25
TCA	10	15	19	11	14	18
Co(II)	15	19	23	17	19	24
Ni(II)	14	18	22	14	17	22
Cu(II)	15	20	24	16	18	23
Miconazole (Standard)	20	25	30	22	24	29

spectrum. This band shows lower shift by 27-32  $\text{cm}^{-1}$  in all complexes, suggesting participation of thiophene  $\nu(\text{C-S-C})$  sulphur in complexation. The appearance of broad band around  $3310 \pm 3 \text{ cm}^{-1}$  in the spectra of all complexes may be due to  $\nu_{\text{stre}}$  of water. A medium intensity band at  $798 \text{ cm}^{-1}$  and  $790 \text{ cm}^{-1}$  respectively in the spectrum of Ni(II) and Cu(II) complexes, are assignable to rocking mode due to coordinated water molecule. The new bands in all complexes at  $470 \pm 8 \text{ cm}^{-1}$  and  $407 \pm 3 \text{ cm}^{-1}$  have been assigned to  $\nu(\text{M-N})$  and  $\nu(\text{M-S})$  modes respectively (Nakamoto, 1998; Neelakantan, 2008)

**Electronic spectra and magnetic moment:** The nature of the ligand field around the metal ion has been deduced from the electronic spectra.

The electronic spectrum of Co(II)-TID complex shows two bands of appreciable intensity at  $12315 \text{ cm}^{-1}$  and  $20040 \text{ cm}^{-1}$  which have tentatively been assigned to  ${}^4\text{A}_2 - {}^4\text{T}_1(\text{F})(\nu_2)$  and  ${}^4\text{A}_2 - {}^4\text{T}_1(\text{P})(\nu_3)$  transition. The magnetic moment is 4.39 B.M. Thus, the tetrahedral geometry has been suggested for this complex. The electronic spectrum of Ni(II)-TID complex show three bands at  $11316 \text{ cm}^{-1}$ ,  $18472 \text{ cm}^{-1}$  and  $23254 \text{ cm}^{-1}$  corresponding to transition  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{1g}(\text{F})(\nu_2)$ ,  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{1g}(\text{F})(\nu_2)$  and  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{1g}(\text{P})(\nu_3)$  respectively. The magnetic moment is 3.11 B.M. These findings are in favor of an octahedral geometry for the Ni(II) complex. The Cu(II)-TID complex shows a broad band at  $14892 \text{ cm}^{-1}$  which is assignable to  ${}^2\text{E}_g - {}^2\text{T}_{2g}$  transition. The magnetic moment is 1.78 B.M. These parameters suggest the octahedral geometry for this Cu(II) complex.

The electronic spectrum of Co(II)-TCA complex shows two bands of appreciable intensity at  $12315 \text{ cm}^{-1}$  and  $20040 \text{ cm}^{-1}$  which have tentatively been assigned to  ${}^4\text{A}_2 - {}^4\text{T}_1(\text{F})(\nu_2)$  and  ${}^4\text{A}_2 - {}^4\text{T}_1(\text{P})(\nu_3)$  transition. The magnetic moment is 4.34 B.M. Thus the tetrahedral geometry has been suggested for this complex. The electronic spectrum of Ni(II)-TCA complex exhibits three bands at  $12300 \text{ cm}^{-1}$ ,  $17412 \text{ cm}^{-1}$  and  $23276 \text{ cm}^{-1}$  which are assignable to  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{1g}(\text{F})(\nu_1)$ ,  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{1g}(\text{F})(\nu_2)$  and  ${}^3\text{A}_{2g}(\text{F}) - {}^3\text{T}_{2g}(\text{P})(\nu_3)$  transition, respec-

tively. The magnetic moment is 3.06 B.M. These findings are in favor of an octahedral geometry for the Ni(II) complex. For the Cu(II)-TCA complex a single broad band at  $14810 \text{ cm}^{-1}$  has been observed, this attributes to  ${}^2\text{E}_g - {}^2\text{T}_{2g}$  transition. Its magnetic moment is 1.86 B.M. Thus the octahedral geometry has been suggested for complex (Mohamed, 2006; Lever, 1984; Sharma, 2010; Chandra, 2009; Dutta, 1993)

**ESR spectra:** The ESR spectra of Cu(II) provide information about the extent of the delocalization of unpaired electron. The X-band ESR spectra of Cu(II) complexes were recorded in the solid state at room temperature and their  $g_{\parallel}$ ,  $g_{\perp}$ ,  $\Delta g$ ,  $g_{\text{av}}$  and  $G$  have been calculated. The values of ESR parameters  $g_{\parallel}$ ,  $g_{\perp}$ ,  $g_{\text{av}}$ ,  $\Delta g$  and  $G$  for Cu(II) complex of TID are 2.0935, 2.1419, 2.1045, 0.14 and 0.8620 respectively.

The exchange coupling interaction between two Cu(II) ions is explained by Hathaway expression  $G = (g_{\parallel} - 2.0023) / (g_{\perp} - 2.0023)$ . According to Hathaway, if the value  $G$  is greater than four ( $G > 4.0$ ), the exchange interaction is negligible; whereas when the value of  $G$  is less than four ( $G < 4.0$ ) a considerable exchange coupling is present in solid complex. The  $G$  values for the complexes are less than four indicate considerable exchange interaction in the complexes (Hathaway, 1987; Mishra, 2005).

**Thermal analyses:** The thermal behavior of metal complexes shows that the hydrated complexes lose molecules of hydration first; followed by decomposition of ligand molecules in the subsequent steps.

The thermogram of  $[\text{Cu}(\text{TID})\text{Cl}_2] \cdot 2\text{H}_2\text{O}$  complex shows a weight loss between  $80-130^\circ\text{C}$  corresponds to the loss of two lattice water molecules from the complex (Remaining wt. %, obs./cal. 93.8/92.19). After  $240^\circ\text{C}$ , a gradual weight loss occurs up to  $360^\circ\text{C}$  indicates the loss of non coordinated part of the ligand in this step (Remaining wt. %, obs./cal. 40.5/39.89). Above this temperature an inflection occurs in curve and the loss in weight continued up to  $650^\circ\text{C}$ . This indicates the elimination of the complex. After this temperature constant weight region due to metal oxide as a final pyrolysis

product appears. (Remaining wt. %, obs./cal. 22.00/21.64) (Al-Shihri, 2003; Wang, 2009; Mohanan, 2010)

The thermal analysis evaluating the thermal stability of the metal complexes, this study also helped to characterize the metal complexes.

**Antimicrobial activities:** The *in-vitro* Antimicrobial activity of the synthesized Schiff base ligands and their corresponding metal complexes on selected bacteria *E. coli* and *S. aureus* and two fungi *A. niger* and *C. albicans* was carried out. All of the tested compounds showed good biological activity against microorganism. On comparing the biological activity of the Schiff base and its metal complexes with the standard bactericide and fungicide, it is show that the metal complexes have moderate activity as compared to the standard but all the complexes are more active than their respective ligands. The higher inhibition zone of metal complexes than those of the ligands can be explained on the basis of Overtone's concept and Chelation theory. On chelation, the polarity of the metal ion will be reduced to greater extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. Further, it increases the delocalization of  $\pi$  - electrons over the whole chelating ring and enhances the penetration of the complexes into lipid membranes and blocking of the metal binding sites in the enzymes of microorganisms. There are other factors which also increases the activity are solubility, conductivity and bond length between the metal and ligand (Singh, 2008; Mishra, 2008; Mohamed, 2009).

The antibacterial and antifungal data are listed in Tables 2 and 3. The investigation of antibacterial data revealed that the Co(II) and Cu(II) complex of displayed highly activity against bactericide and fungicide.

## CONCLUSION

In the present research studies, our efforts are synthesized of some newly compounds from the conventional as well as microwave methods. These synthesized compounds characterized by various physicochemical and spectral analyses. Thermogravimetric studied of the complexes also helped to characterize of the complexes. The antimicrobial data show that the metal complexes to be more biological active compared to those parent Schiff base ligand against all pathogenic species. The compounds also inhibit the growth of fungi and bacteria to a greater extent as the concentration is increased.

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