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Synthesis of New Mn(II), Co(II) and Cu(II) Complexes Grabbed in Novel Functionalized Ionic Liquid Tagged Schiff base: Physico-chemical Properties and Antibacterial Applications

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Abstract

An ionic liquid tagged schiff base, [1-{2-(2-hydroxybenzylideneamino)ethyl}-3-methylimidazolium]PF_6 was synthesized from a functionalized ionic liquid (FIL) 2-aminoethyl-3-methylimidazolium hexafluorophosphate, [2-aemim]PF_6 and salicylaldehyde without using any organic solvent. The Schiff base was used as a ligand to trap Mn(II), Co(II), Cu(II) ions from its corresponding metal acetates and to form the new transition metal complexes. The synthesized Mn(II), Co(II), Cu(II) complexes along with the Schiff base were characterized by ESI-MS, PXRD, UV-Visible, FT-IR, ^{1}H NMR spectroscopic and analytical (elemental analysis and magnetic susceptibility measurements) methods. Based on these spectral analysis data tetra coordinated geometries were assigned for the newly synthesized metal(II) complexes. Antibacterial sensitivity of the Schiff base and the Mn(II), Co(II), Cu(II) complexes were evaluated against gram positive (*Staphylococcus aureus*) and gram negative (*Escherichia coli*) bacteria to assess their minimum zone of inhibition.

Keywords: Ionic liquid; Schiff base; Metal complexes; Antibacterial activity

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1. Introduction

The ongoing chemical research is turned into an essential need to develop green chemistry process to avoid the noxious materials and useless waste generation. Ionic liquids (ILs) are viewed as the new, safe and ecofriendly organic solvents with a very wide liquidus range [1,2]. The reason for the increased interest in last few decades due to the realization that these organic salts have greater utility as solvents for reactions and material processing, as extraction media or as working fluids in mechanical applications and so on [3-5]. Physical properties of ionic liquids such as viscosity, hydrophobicity, melting point, and solubility can be fine-tuned by changing the anions and altering the length of the alkyl groups on the cations [6]. In reality, most ionic liquids are also exists in liquid state at room temperature and termed as room temperature ionic liquids (RTIL). ILs is usually composed of an organic cation

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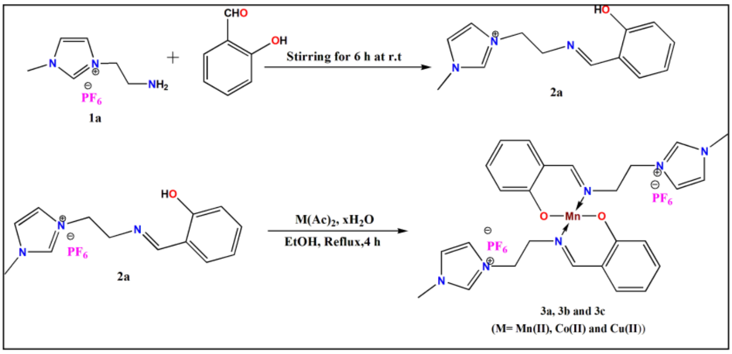
and inorganic or organic polyatomic anion. Since there are lots of known and potential cations and anions, the potential number of ionic liquids is huge but designing their geometry and determining functionality as a green solvent requires much more substantial investigation of physical and chemical properties [7,8]. The great variety of ILs can also be enhanced remarkably by incorporating effective functional groups into the structure so-called functionalized ionic liquids (FILs) to enhance the versatility of ILs such as the property to interact with metal centres and solubilise metal ions for catalytic and electrochemical applications [9,10].

Schiff bases are very significant in the field of coordination chemistry as well as medicinal and pharmaceutical field. A wide range of biological applications including antibacterial [11,12], antifungal [13] and antitumor activity [14] were investigated not only with the Schiff bases but their metal complexes also. Microbes encounter a variety of metal ions in the environment and interact with them which are sometimes beneficial or detrimental depending on the chemical/physical nature and oxidation state of the metal ion. Of particular interest are those metal ions, which exist mostly as cations (or cationic complexes), oxyanions, as salts or oxides in crystalline form or as amorphous precipitates in insoluble form. The microbes have the ability to bind to metal ions present in the external environment at the cell surface and to transport them into the cell for various intracellular functions [15]. Salicylaldehyde and its derivatives are useful carbonyl precursors for the synthesis of a large variety of Schiff bases with wide variety of interesting properties. The systematic research towards the invention of new ligand, i.e., replacing the metal ion of the complex systematically is essential to obtain not only the basic knowledge of the coordination chemistry but also an advanced property. Hence in this article, an attempt was taken to synthesize an ionic liquid tagged Schiff base 1-{2-[(2-hydroxybenzylidene)amino]ethyl}-3-methylimidazolium hexafluorophosphate from functionalized ionic liquid (FIL) 2-aminoethyl-3-methylimidazolium hexafluorophosphate, [2-aemim]PF_6 and salicylaldehyde without any organic solvent and the Mn(II), Co(II) and Cu(II) complexes from the corresponding metal acetates (Scheme 1). The synthesized compounds were characterized by various analytical techniques and screened their biological activities against gram positive / negative bacteria.



Scheme 1. Synthesis of the ligand, 1-{2-[(2-hydroxybenzylidene)amino]ethyl}-3-methylimidazolium hexafluorophosphate (2a) and the M(II) complexes (3a, 3b and 3c) from (2a).

2. Experimental Section

2.1. *Materials and methods*

1-methyl imidazole, potassium hexafluorophosphate and 2-bromoethylamine hydrobromide were purchased from Sigma Aldrich, Germany. Salicylaldehyde, Metal acetates and all other chemicals were obtained from SD fine Chemicals, India. The required solvents methanol, petroleum ether, chloroform, DMF and DMSO were used after purification by the standard methods describe in the literature.

Elemental micro-analyses (C, H and N) were performed by using Perkin–Elmer (Model 240C) analyzer. Metal content was determined with the aid of AAS (Varian, SpectrAA 50B) by using standard solution from Sigma-Aldrich, Germany. ESI-MS, Mass spectra was recorded on a Agilent 1100 LC equipped with an MSD trap. The purity of the Schiff base and its complexes were confirmed by thin layer chromatography (TLC) on silica gel plates and TLC visualization was done by UV-light and iodine. Powder X-ray diffraction (XRD) data obtained from INEL XRD Model Equinox 1000 using Cu Kα radiation (2θ= 0-90˚). FT-IR spectra were recorded in KBr pellets with a Perkin-Elmer Spectrum RX-I FT-IR spectrometer, operating in the region 4000 to 400 cm^{-1}. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a FT-NMR BRUKER ADVANCE II 400 MHz and 100 MHz spectrometer respectively by using DMSO-d_6 and D_2O as solvent at room temperature. Melting points were recorded by open capillary method. Antibacterial activities (*in vitro*) of the synthesized ligand and the complexes were studied by disc diffusion method against commonly known bacteria, *viz.*, *Staphylococcus aureus* and *Escherichia coli* with respect to the standard drug Ampicilin.

2.2. *Synthesis of [1-(2-aminoethyl)-3-methyllimidazolium hexafluoro phosphate,(1a) [2-aeeim] PF_6*

1-methylimidazole (0.05 mol) and 2-bromoethylamine hydrobromide (0.05 mol) in 25 mL of acetonitrile were heated with stirring at 80 °C for 6 h. On completion, the solvent was removed by distillation. Then KPF_6 (0.05 mol) was added in 20 mL of CH_3CN/H_2O (1:1, v/v) to the reaction mixture. The solution was kept for 24 h at room temperature and NaOH (0.05 mol) was added for neutralization. Solvents were evaporated under vacuum. The obtained yellow oil was washed successively with chloroform (10 mL × 3) and ether (10 mL × 3). After drying for 6 h under vacuum at 80 °C, the ionic liquid was obtained [16].

Yellow oil; 9.35 g. FT-IR (KBr, cm^{-1}): 3429 (N-H), 3106, 2365, 1617 (C=N), 1175, 846 (P-F). ^1H NMR (400 MHz, D_2O) δ= 3.26 (t, 2H); 3.83 (s, 3H), 4.44 (t, 1H), 7.39 (s, 1H), 7.48 (s, 1H), 8.52 (s, 2H), 8.79 (s, 1H); ^{13}C-NMR (100 MHz, D_2O) δ= 136.87, 124.53, 122.97, 53.35, 36.01, 27.88. ESI-MS (CH_3OH, *m/z*): 126.20 [$(M-PF_6)^+$]. Anal. Calcd. for $C_6H_{12}F_6N_3P$ (271): C, 26.58; H, 4.46; N, 15.50. Found: C, 26.42; H, 4.39; N, 15.46 %

2.3. *Synthesis of imidazolium ionic liquid-supported Schiff base, 1-{2-[(2-hydroxy benzylidene)amino]ethyl}-3-methylimidazolium hexafluorophosphate (2a)*

A mixture of salicylaldehyde (1.22 g, 10 mmol) and [2-aemim]PF_6 (2.71 g, 10 mmol) was stirred without any solvent at room temperature for 6 h. After completion of the reaction, as indicated by TLC, the reaction mixture was diluted with methanol, MeOH (10 mL). The precipitate was then filtered, washed with cold ethanol and dried to afford the expected ligand as a pale yellow solid.

Yellow mass; M. P. 130-133 ºC; ^1H NMR (400 MHz, DMSO-d6): δ= 3.40 (s, 3H), 4.52 (t, 2H), 4.63 (t, 2H), 7.71–7.89 (m, 4H), 7.94 (s, 1H), 8.07 (s, 1H), 9.17 (s, 1H), 9.31 (s, 1H), 8.23 (s, 1H). ^{13}C-NMR (100 MHz, DMSO-d6): δ = 135.95, 135.87, 133.92, 123.00, 122.95, 122.40, 121.45, 119.55, 53.39, 52.69, 50.82, 38.89, 35.46. Anal. Calcd. for $C_{13}H_{16}N_3OPF_6$ (375): C, 41.61; H, 4.30; N, 11.20. Found: C, 40.87; H, 4.21; N, 11.09 %. FT-IR (KBr, cm^{-1}): 3428 (O-H), 1639 (C=N), 1276 (C-O), 835 (P-F). ESI-MS (CH_3OH, m/z): 230 ($[M-PF_6]^+$, M= $[C_{13}H_{16}N_3O]^+$). UV-Vis (Methanol) λmax/nm: 217, 245, 334.

2.4. *Synthesis of metal complexes (**3a**, **3b** and **3c**)*

To a solution of ligand, (0.50 g, 1.30 mmol), in ethanol, EtOH (20 mL) solution of ethanolic metal acetate Mn(II), Co(II) and Cu(II)), *viz.*, (0.65 mmol) was added. The reactants were refluxed for 6 h until the starting materials were completely consumed as monitored by TLC. On completion of reaction, solvents were evaporated and the mixture was cooled to room temperature. The precipitate was collected by filtration, washed gradually with cold ethanol. The yield was dried in vacuum desiccators to obtain the solid mass. The complexes are soluble in *N*, *N*-dimethylformamide, dimethylsulphoxide, and methanol.

2.4.1. *Mn(II) complex (**3a**)*

Brown solid; M. P.: 162-164 ^{o}C; FT-IR (KBr, cm^{-1}): 3446 (O-H), 1633 (C=N), 1278 (C-O), 837 (P-F), 621 (M-O), 556 (M-N). UV-Vis (Methanol) λmax/nm: 211, 252, 319. ESI-MS (CH_3OH, m/z): 515 ($[M+2H-2PF_6]^+$, M= $[C_{26}H_{30}MnN_6O_2]^+$). Anal. Calc. for $C_{26}H_{30}MnF_{12}N_6O_2P_2$ (803.42): C, 38.87; H, 3.76; N, 10.46; Mn, 6.84. Found: C, 38.56; H, 3.66; N, 10.42, Co, 6.49 %.

2.4.2. *Co(II) complex (**3b**)*

Dark brown solid; M. P. 155-157 ^{o}C; FT-IR (KBr, cm^{-1}): 3425 (O-H), 1638 (C=N), 1311 (C-O), 843 (P-F), 623 (M-O), 557 (M-N). UV-Vis (Methanol) λmax/nm: 210, 249, 334, 390. ESI-MS (CH_3OH, m/z): 516 ($[M-H-2PF_6]^+$, M=$[C_{26}H_{30}CoN_6O_2]^+$). Anal. Calc. for $C_{26}H_{30}CoF_{12}N_6O_2P_2$ (807.42): C, 38.68; H, 3.75; N, 10.41; Co, 7.30. Found: C, 38.51; H, 3.69; N, 10.32, Ni, 7.13 %.

2.4.3. *Cu(II) complex (**3c**)*

Dark green solid; M. P. 166-169 ^{o}C; FT-IR (KBr, cm^{-1}): 3427 (O-H), 1634 (C=N), 1313 (C-O), 841 (P-F), 621 (M-O), 557 (M-N). UV-Vis (Methanol) λmax/nm: 217, 337, 397. ESI-MS (CH_3OH, m/z): 524 ($[M+3H-2PF_6]^+$, M= $[C_{26}H_{30}CuN_6O_2]^+$). Anal. Calc. for $C_{26}H_{30}CuF_{12}N_6O_2P_2$ (812): C, 38.46; H, 3.72; N, 10.35; Cu, 7.83. Found: C, 38.21; H, 3.69; N, 10.15; Cu, 7.56 %.

2.4. *Antibacterial activity*

The newly synthesized metal complexes (**3a**, **3b**, **3c**) and the ligand (**2a**) were tested against the gram-negative bacterium *Escherichia coli* ATCC 69905 and the gram-positive bacterium *Staphylococcus aureus* ATCC 6633. The *in vitro* biological screening effects of the newly synthesized compounds were tested by disc diffusion method using Muller-Hinton agar medium (Difco) at a pH 7.2. In a disc diffusion method, 24 h old fresh cultures of the tested organism were spread on different MH plate with the help of sterile spreader [17,18]. Petri dishes containing the tested organisms were placed in an incubator at 37 ^{o}C for 24 h. Fresh stock solutions of the ligand and complexes were prepared by dissolving in DMSO and serial dilutions of the compounds were made in sterile distilled water. The concentrations of the compounds were 100, 250 and 500 µg/mL. Standard drug Ampicilin were used as a positive control and the solvent (DMSO) were used as negative control.

3. Results and Discussion

FT-IR spectra of the ligand (**2a**) showed a band corresponding to the azomethine group (-C=N) at 1639 cm^{-1}. On complexation, this band shifted to lower wave number range of 1633-1638 cm^{-1} which suggested the involvement of N- atom of azomethine (-C=N) group in the complex formation [19]. A strong broad absorption band observed at 3428-3150 cm^{-1}; which was appeared due to the hydrogen bonded -OH of the phenolic group with H–C(=N) group of the ligand (OH…N=C) [20,21]. For the complexes, (**3a**, **3b** and **3c**) broad band observed at 3425-3448 cm^{-1}, may be assigned to the presence of the hydrated water molecules. The band for phenolic C-O of free ligand was assigned at 1276 cm^{-1}. Upon complexation, this band was moved to higher wave number 1278-1313 cm^{-1} for all metal complexes [22]. This appearance of M-O band at 621-623 cm^{-1} was identified in the spectra of the metal complexes. This interpretation was further emphasized by the presence of a new weak to medium intensity absorption in the region 556-559 cm^{-1} that may be attributed to M-N stretching vibration for the metal complexes [23]. The bands in the range of 835-843 cm^{-1} in the spectra of the ligand and metal complexes were assigned for P-F stretching frequency.

The mass spectra of the ligand showed a molecular ion peaks (*m/z*) at 230, which was assigned for M+, $[C_{13}H_{16}N_3O]^+$ peak. A peak (*m/z*) at 515 which was due to $[M\text{-}2H\text{-}2PF_6]^+$ of Mn(II) complex (**3a**) (M= $[C_{26}H_{30}MnN_6O_2]^+$) ion. The ion peck for Co(II) complex (**3b**) (*m/z*) at 517 in the ESI-MS spectrum was assigned to $[M\text{-}2PF_6]^+$, (M= $[C_{26}H_{30}CoN_6O_2]^+$) ion. The Cu(II) complex (**3c**) exhibited a peak (*m/z*) at 524, for $[M+3H\text{-}2PF_6]^+$ (M= $[C_{26}H_{30}CuN_6O_2]^+$) ion. The different molecular ion peaks, appeared in the mass spectra of the complexes, were attributed to different fragmentations of the metal complexes by successive rupture of different bonds in order to form stable ions.

^{1}H-NMR of the LH (**2a**) showed singlet at 9.17 ppm assignable to proton of the azomethine group (-CH=N-) presumable due to the effect of the *ortho*-hydroxyl group in the aromatic ring. A singlet at 8.23 ppm can tentatively be assigned to hydroxyl proton. The LH (**2a**) displayed downfield shift of the –OH proton was due to intermolecular (O-H...N) hydrogen bond [23]. ^{13}C-NMR spectra of ligand exhibited peaks at δ 135.95 and 135.87 presumably due to the phenolic (C-O) and imino (-CH=N) carbon atoms (due to Keto-imine tautomerism). The chemical shifts of the aromatic carbons appeared at δ 123.60, 122.95, 122.40, 121.45 and 119.55.

The PXRD pattern of ligand (**2a**) displayed sharp peaks probably due to crystalline nature. The spectra of the Mn(II), Co(II) and Cu(II) (**3a, 3b** & **3c**) complexes didn't show sharp peaks might be due to their amorphous nature The crystalline sizes were determined using the Debye Scherer's equation: $D= 0.9\lambda/\beta\cos\theta$, in which constant 0.9 is the shape factor, λ indicates the X-ray wavelength (1.5406 Å), β is the full width at half maximum (FWHM) and θ denotes the Bragg diffraction angle. The experimental average grain sizes of Schiff base (**2a**) and the metal complexes were calculated to be 79.44 nm (**2a**), 42.23 nm (**3a**), 32.21 nm (**3b**) and 39.52 nm (**3c**).

UV-Visible spectra of the ligand and the metal complexes (Fig. 1) were conducted at room temperature using methanol as solvent. The electronic spectrum of LH (**2a**) showed three absorption bands at 334, 245 and 217 nm due to n→π*, π→π* and transitions involved with the imidazolium moiety, respectively [24, 25]. The three bands at 319, 252 and 211 nm were appeared. The ligand band at 334 nm showed a hypsochromic shift probably for coordination with Mn^{2+} (d^5) ion. For the complexes, the bands that appeared below 352 nm were ligand centred transitions (n→π* and π→π*). The electronic spectra of the Co(II) complex (**3a**) exhibited three bands at 334, 249 and 210 nm and a shoulder around 390 nm. The bands were assigned to $^4A_2 \rightarrow {}^4T_1(P)$, $^4A_2 \rightarrow {}^4T_1(F)$ and $^4A_2 \rightarrow {}^4T_2$ transitions, respectively. The experimental magnetic moment values were 5.79 and 3.87 B.M. for **3b** and **3c** respectively**.** So, The UV-Vis spectra along with the magnetic moment values of Mn(II) complex and Co(II) complex proposed tetrahedral geometry for both the cases [26]. UV-visible spectra of Cu(II) complex (**3c**) showed d→π* metal-ligand charge transfer transition (MLCT) in the region 397 nm had been assigned to the combination of $^2B_{1g} \rightarrow {}^2E_g$ and $^2B_{1g} \rightarrow {}^2B_{2g}$ transitions in a distorted square planar environment [27,28]. The observed magnetic moment for Cu(II) complex (**3c**) was 1.81 B.M. consistent with the presence of an unpaired electron.

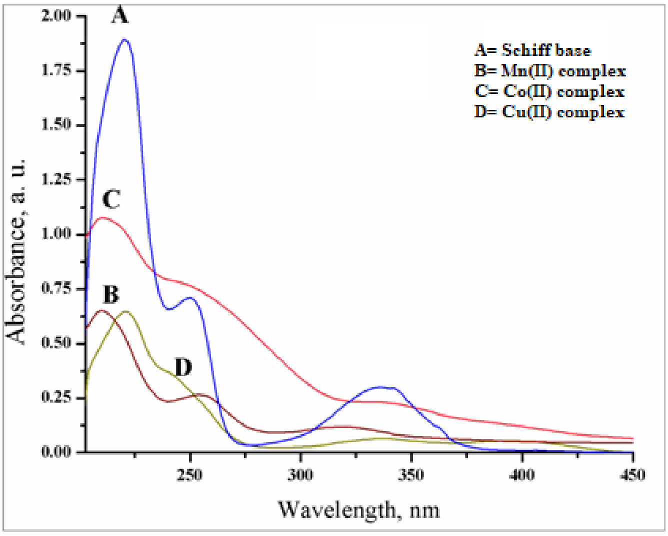


Fig. 1. UV-Visible spectra of the ligand (2a) and its metal complexes (3a, 3b and 3c).

The newly synthesized metal complexes (**3a**, **3b** and **3c**) along with the ligand (**2a**) screened against the gram negative bacteria *Escherichia Coli* and gram positive bacteria *Staphylococcus aureus* are shown in Fig. 2. The zone of inhibition values for each compounds were measured in millimetres and the values are reported in Table 1 as a mean of three replicate. The inhibition zone values were used to determine the antibacterial efficiency of the tested compounds. The result suggested that all the synthesized materials have little or moderate antibacterial activity against the gram negative bacteria *Escherichia coli* as compared to standard drug Ampicilin. The result also indicated that the compound, the ligand (**2a)** has good activity against the gram positive bacteria *Staphylococcus aureus* as compared to standard drug in all the studied concentration range and further the antibacterial activity is enhanced as the concentration of compound was increased. The Cu(II) complex (**3c**) also showed good activity against *Staphylococcus aureus* only at the concentration 500 µg/mL. However, other complexes have little or moderate activity as compared to standard drug Ampicilin.

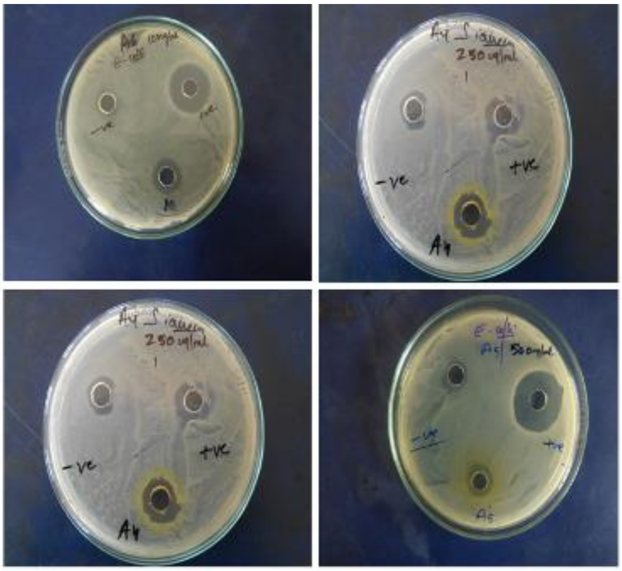


Fig. 2. MIC of the ligand (2a) and its metal complexes (3a, 3b and 3c)

Table 1. Antibacterial activity of Schiff base (2a) and its metal complexes (3b, 4b and 5b) against bacterial strains with their minimum zone of inhibition and MIC (µg/mL) values.

Compound	Test organism	100 µg/mL			250 µg/mL			500 µg/mL		
		+	-	Test	+	-	Test	+	-	Test
LH (2a)	*E.coli*	15	-	-	18	-	12	23	-	15
	S. aureus	-	-	14	14	-	17	-	-	21
Mn(II) complex (3a)	*E.coli*	-	-	13	20	-	11	21	-	15
	S. aureus	-	-	-	13	-	-	15	-	-
Co(II) complex (3b)	*E.coli*	18	-	15	22	-	17	25	-	13
	S. aureus	-	-	-	12	-	11	13	-	12
Cu(II) complex (3c)	*E.coli*	17	-	14	21	-	-	25	-	15
	S. aureus	-	-	-	11	-	11	11	-	13

4. Conclusion

In this study, the synthesis, characterization and antibacterial sensitivity of the synthesized novel ionic liquid tagged Schiff base and its Mn(II), Co(II), Cu(II) complexes are reported. The complexes were formed in 1:2 (metal: ligand) ratio as confirmed by the spectral and analytical data analysis. The Schiff base ligand played as a bidentate ligand and coordinated to metal ions through phenolic oxygen and the azomethine nitrogen. The results of different analytical and spectroscopic data showed that the ligand was oriented in tetra-coordinated geometries around the metal centre. The biological importance of the synthesized compounds was also unfolded through screening its antibacterial responses. The synthesized Schiff base and the Cu(II) complex showed significant antibacterial activities where as the Mn(II) and Co(II) complexes exhibited minimum activity against the two commonly known bacteria, *viz.*, *Staphylococcus aureus* and *Escherichia coli.*

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