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Synthesis, spectroscopic studies and pesticidal activity of transition metal complexes with unsymmetrical schiff base

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Unsymmetrical Schiff base glyoxal salicylaldehyde succinic acid dihydrazide (gssdh), and their Co(II), Ni(II), Cu(II), and Zn(II) complexes have been synthesized and analyzed. These complexes' structures are explained based on magnetic, electronic, IR, and NMR (1H and 13C) spectral studies. These ligands show monobasic tetra-dentate behavior, bonding through C=O, two C=N, and a phenolate group. Ni(II) complex show square planar geometry while Cu(II) and Co(II) complexes exhibit tetrahedral geometry in the solid-state shown by electronic spectra. However, Co(II) and Cu(II) complexes adopt octahedral geometry in DMSO solution. The insecticidal activity of these compounds is tested, and compared their activity with their standard. Synthesized compounds show moderate insecticidal activity at higher concentrations. At low concentrations, these compounds have anti-fungal and antibacterial properties but do not show the pesticide property.

Keywords: Antibacterial, Insecticidal activity, Magnetic

Schiff bases are prepared by the reaction of primary amine and aldehyde or ketone. Schiff's base name came into existence after the scientist name Hugo Schiff. There are huge numbers of Schiff bases discovered and investigated till now, but unsymmetrical Schiff bases are very few in numbers. The geometry of these molecules makes them more interesting and applicable due to the availability of different binding modes. Pesticides are chemical protection of plants, based on the use of several organic and inorganic compounds; toxic to harmful organisms. The pesticide is particularly effective in controlling harmful pests to improve the quality and quantity of agricultural production[1]. These agents are also used to preserve food grains from pests. For this purpose, several thousand chemical agents have already been formulated. These possess all the characteristics of an ideal pesticide. Maximum of these kill the useful germs, not the target pests; organisms useful in restoring the fertility of the soil. Further, many of these are not biodegradable in nature. After application, these are most often found to exist in sublethal doses in the environment for a long time. There are tremendous applications of Schiff bases like pigments, catalysts, intermediates in organic synthesis, dyes, redox properties[2], and polymer stabilizers[3]. A number of Schiff bases possess biological activities[4], *i.e.*, Antifungal, antibacterial[5], antipyretic properties, anti-inflammatory, antimalarial[3,6], anticancerous[7] and antiviral[8]. However, unsymmetrical Schiff base ligand has been synthesized by the condensation of two different keto group-containing compounds (glyoxal and salicylaldehyde) with a dihydrazide (succinic acid dihydrazide) and used to prepare their metal complexes under certain specific conditions. These compounds are examined against various pests and found as biologically active compounds. Complexes showed very exciting structural diversities of metal chelates. We have synthesized Co(II), Ni(II), Cu(II), and Zn(II) complexes of gssdh ligand.

Material and Methods

Materials

Commercially available reagents have been used without further purification, and all experiments have been carried out in the open condition. The metal salts, glyoxal, salicylaldehyde, and solvents were purchased from SD Fine Chemicals, India, and used. Diethyl oxalate, diethyl malonate, and hydrazine hydrate (Merck Chemicals, India) are used in synthesis. Succinic acid dihydrazide has been prepared according to a reported method[9-11] by treatment of hydrazine hydrate with diethyl succinate.

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Synthesis of Ligand

Glyoxal salicylaldehyde succinic acid dihydrazone (gssdh) has been synthesized by reacting 80 mL aqueous solution of succinic acid dihydrazide (10 mmol) with 50 mL aqueous ethanolic solution of a mixture containing glyoxal (10 mmol, 0.58 mL) and salicylaldehyde (10 mmol, 1.22 mL) in a round bottom flask. The reactants have been stirred on a magnetic stirrer continuously for 5-6 hours at room temperature. A cream-colored precipitate of the product has been obtained; it has been filtered and washed with aqueous ethanol (*v/v*, 1:1). The pure ligand has been dried in a desiccator over anhydrous calcium chloride at room temperature. Cream yellow, yield (60%). Ligands are highly insoluble in organic solvents, So there is no possibility of forming crystals. IR data for the ligands are (ν cm-1, KBr): ν(OH) 3380b; ν (NH) 3220b; ν(C=O) 1667b; ν (CN) 1642s and ν (N-N) 967w. NMR data of the ligand are:1HNMR signals(ppm) CH2 (3.85 s), Aromatic protons (6.70-7.35 m), HN (7.57 s, 7.68s), -CH=N (5.55 s, 5.8d), Ph-OH (5.3 s) as shown in (Fig. 1); and carbon 13 NMR signals for the ligand are represented in (Fig 2); 13C NMR signals (ppm) CH2(42.3), Aromatic carbon (116.9-132.6), C-OH (148.9), -CH=N (160.6, 157.9)

Synthesis of the metal complexes:

The Co(II), Ni(II), Cu(II), and Zn(II) complexes of gssdh ligand have been synthesized by reaction of ethanolic solutions of each metal(II) chloride with 50 mL ethanolic slurry solution of the powdered ligands gssdh (10 mmol, 2.62 g) in 1:1 (M:L) molar ratio in a round bottom flask keeping the amount of metal salts in slight excess than the required amount (10 mmol). The reaction mixture has been refluxed for 4 hours for Cu(II) and Zn(II) complexes and 6 hours for Co(II) and Ni(II) complexes of the ligand with shaking at regular intervals. The solid products have been obtained and are filtered, washed thoroughly with ethanol to remove the un-reacted metal chloride and dried in a desiccator at room temperature (Fig. 3).

1. Co(gssdh-H)]Cl. Green, yield (60%). M.p. >300 IR (ν cm^{-1}, KBr): ν (NH) 3213b; ν (C=O) 1669b, 1650m; ν (C=N) 1615s; ν (C-O^{-}) 1271s; ν (N-N) 1002m, ν (M-O) 533m; ν (M-N) 474m.
2. [Ni(gssdh-H)]Cl. Orange, yield (63%). M.p. >300, ν (NH) 3208b; ν (C=O) 1667b, 1647m; ν (C=N) 1617s; ν (C-O^{-})1271s; ν (N-N) 1003w, m(M-O) 534w; ν (M-N) 474m.
3. [Cu(gssdh-H)]Cl. Dark brown, yield (61%). M.p. >300 ν (NH) 3213b; ν (C=O) 1669b, 1651m; ν (C=N) 1613w; ν (C-O^{-}) 1273s; ν (N-N) 1003w, ν (M-O) 522w; ν (M-N) 472w.
4. [Zn(gssdh-H)]Cl. Yellow, yield (66%). M.p. >300, ν (NH) 3210b; ν (C=O) 1668b, 1649s; ν (C=N) 1616m; ν (C-O^{-}) 1270m; ν (N-N) 1001m, ν (M-O) 533w; ν (M-N) 479w.

^{1}HNMR signals (ppm) CH$_2$ (3.90 s), Aromatic protons (6.72-7.80 m), HN (7.80 s, 7.65s), -CH=N (5.6s, 5.90d) and ^{13}C NMR signals for the complex are:

^{13}C NMR signals (ppm) CH$_2$ (42.8), Aromatic carbon (120-130.6), C-OH (150.1), −CH=N (161.6, 159.1)

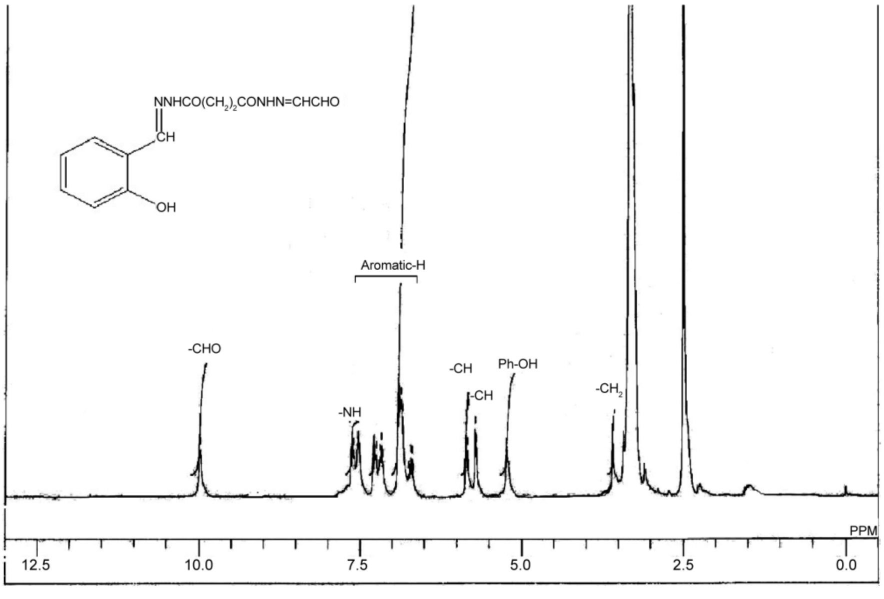

Fig. 1. — ^{1}H NMR spectra of ligand gssdh

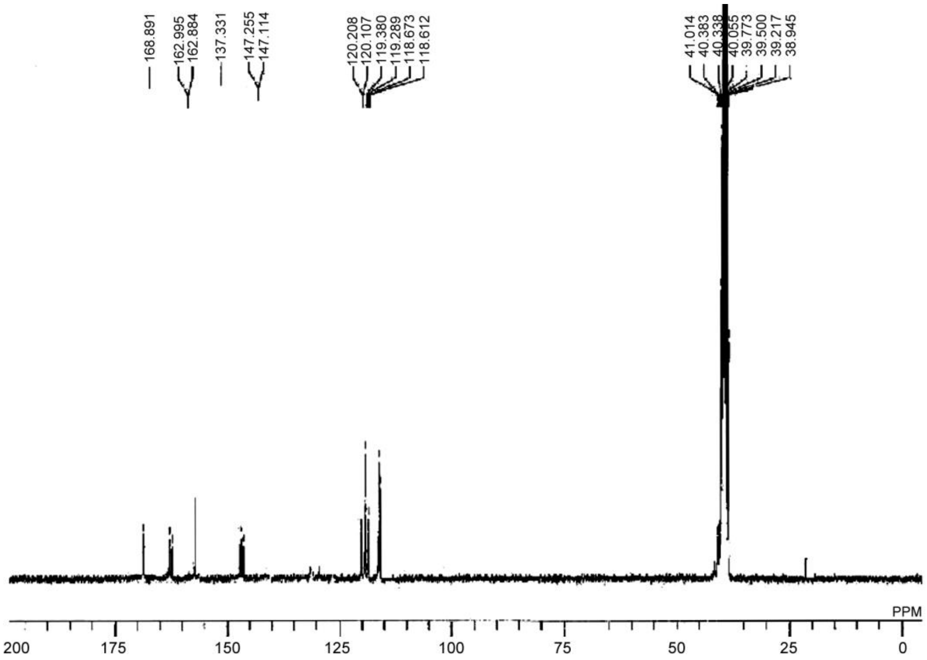

Fig. 2 — ^{13}C NMR spectra of ligand gssdh

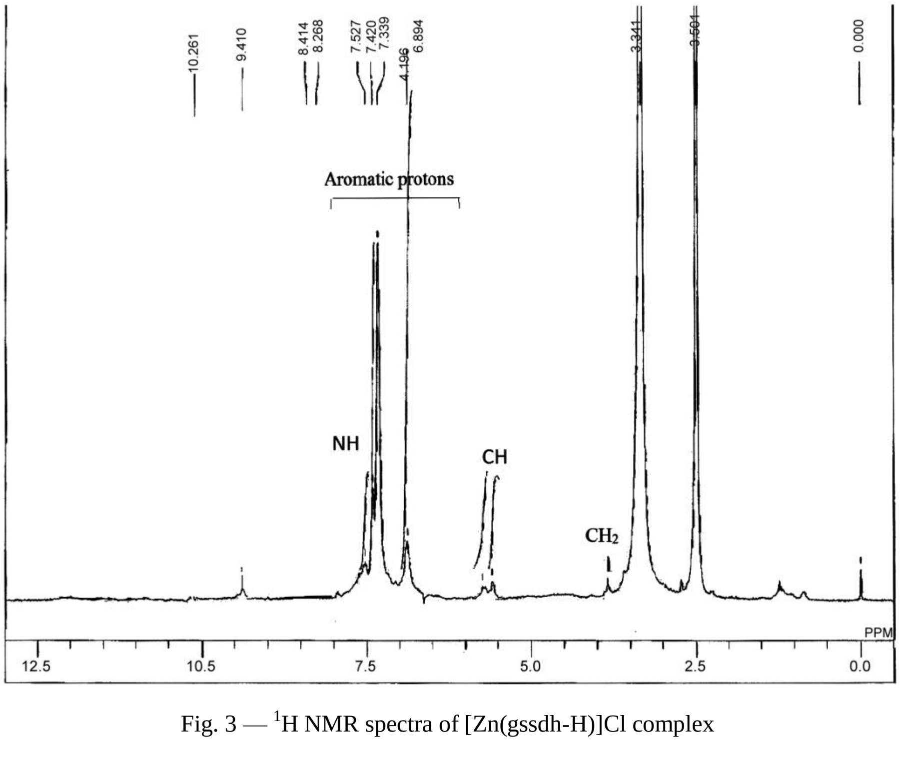

Fig. 3 — ^{1}H NMR spectra of [Zn(gssdh-H)]Cl complex

Physico-chemical measurements

The metal and chloride contents are analyzed gravimetrically by applying standard literature methods[12]. Magnetic susceptibility measurements are done at room temperature on a Cahn-Faraday balance using $Hg[Co(SCN)_4]$ as the calibrant. The complexes electronic spectra have been analyzed on a Shimadzu spectrophotometer, model, Pharmaspec UV-1700 in nujol, and DMSO solution. Infrared spectra are recorded in KBr on a Varian 3100 FT-IR spectrophotometer in 4000-400 cm^{-1} region. ^{1}H and ^{13}C NMR spectra of the ligands have been recorded in

DMSO-d6 on a JEOL AL-300 FT-NMR multinuclear spectrometer. Tetramethylsilane (TMS) as an internal standard is used, and Chemical shifts are reported in parts per million (ppm).

Biological activity

Antifungal activity

We have checked the antifungal activity against the fungi *Aspergillus* sp. This species has been collected from the host plants' contaminated organs on potato dextrose agar medium. A single spore isolation technique has purified the cultures of the fungi. The solution in different concentrations of 0.5, 1, and 1.5 mg/mL of each compound in DMF+ethanol is made ready for testing spore growth and germination. A drop of this solution of each concentration is kept separately on glass slides. The conidia, fungal reproducing spores (approximately 200) lifts with an inoculating needle, are mixed to compound separately. Each treatment are replicated thrice, and a parallel DMF + ethanol solvent control set is run simultaneously on each glass slides. All these slides have been incubated in humid chambers at 25 ± 2C for 24 h. Each slide is observed under the microscope for spore germination, and percent germination are finally calculated. The results have also been evaluated with a standard antifungal drug Miconazole at the same concentrations.

Antibacterial activity

The ligand and its complexes have shown antibacterial activity and have been studied against *E. coli* bacteria. Each of these compounds is dissolved in DMSO, and solutions of the concentration 3 mg/mL and 1.5 mg/mL have been prepared separately. Whatman filter paper (No. 42) discs of uniform diameter (2 cm) are cut and sterilized in an autoclave. The paper discs are saturated in the desired concentration of the complex solutions. They have placed aseptically in the Petri dishes with nutrient agar media (agar 20 g + beef extract 3 g + peptone 5 g) seeded with E. coli bacteria separately. The Petri dishes are incubated at 37°C, and the inhibition zones are recorded after 24 h of incubation. Each treatment is repeated five times. The everyday standard antibiotic Ampicillin's antibacterial activity is also recorded using the same procedure as above at the same concentrations and solvent. Evaluation of minimum inhibitory concentration (MIC) value: The antibacterial protecting concentrations of the compounds used have been estimated from the minimum inhibitory concentration (MIC) value. The MIC is determined using the disc diffusion technique by preparing discs containing 0.1-1.0 mg/mL of each compound against both the bacteria and following the protocol. All the compounds are more effective at 1.5 and 3.0 mg/mL concentrations. Therefore, all the compounds have been tested at these concentrations against the bacteria.

The antibacterial screening concentrations of the compounds to be used are estimated from the minimum inhibitory concentration (MIC) value. The MIC is determined using the disc diffusion technique by preparing discs containing 0.1-1.0 mg/mL of each compound against the bacteria and applying the protocol.

Results and Discussion

It has been clear from the analytical data that the ligand, gssdh are formed by unsymmetrical condensation of glyoxal and salicylaldehyde with succinic acid dihydrazide. The ligand deprotonate their phenolic protons during complexation with metal(II) chloride and form 1:1 (M:L) complexes of general composition [M(gssdh-H)]Cl. The reactions may be written as:

$MCl_2.xH_2O$ +gssdh → [M(gssdh-H)]Cl + HCl + xH_2O

where, M = Co(II), Ni(II), Cu(II) and Zn(II)

Most of the metal complexes are coloured amorphous solids. The Co(II) complex is reddish green but changes to red on addition of water or on dissolving in DMSO. Similarly, Cu(II) complex are dark brown and turn green in water or DMSO solution. This behaviour of colour change is probably because Co(II) and Cu(II) complexes increase their coordination number from four to six in presence of water and DMSO solution. However, the Ni(II) complex gssdh do not show colour variation under similar conditions, and are yellow. The ligands and their metal complexes are insoluble in water and common organic solvents such as ethanol, methanol, chloroform, benzene, DMF and diethyl ether.

Magnetic moment and Electronic spectra:

Copper(II) complex, show μeff value 1.8 B.M. corresponding to one unpaired electron. In solid state (nujol medium), the copper(II) complexes of gssdh show three bands at 630, 465, 395 nm. These bands correspond to $^2B1g \rightarrow {}^2A1g$, $\rightarrow {}^2B2g$ and $\rightarrow {}^2Eg$ transitions, indicating a square planar geometry for the complexes[12] (Table 1).

In DMSO solution, however, a broad band has been observed at 680 nm, gssdh complexes, suggesting a distorted octahedral geometry. The [Ni(gssdh-H)]Cl complex shows three electronic spectral bands at 818, 478 and 354 nm, indicates a square planar geometry in solid state. Ni (II) complex show diamagnetic behaviour in their complex. The μ_{eff} value observed at 4.35BM for [Co(gssdh-H)]Cl complex indicates three unpaired electrons. In solid state Co(II) complex shows two bands at 680 and 378 nm in solid state exhibits tetrahedral geometry. While in DMSO Solution it shows three bands at 1065, 520 and 436 nm that indicates its octahedral geometry,

Infra Red spectra

The bonding between metals and ligand have been clearly shown by comparing IR spectra of ligand and their metal complexes. Some significant band are selected to observe that either complex formation has occurred or not and which bonds are involved in complex formation. The ligand show broad band at 3380 cm^{-1} due to ν(OH) and at 3220 cm^{-1} for ν(NH). In metal complexes IR band for NH group either occur at same frequency or slightly shifted. It confirms that NH group is not participated in bond formation with metal[14]. ν(OH) band is absent in all the metal complexes; denotes that OH band is involved in bonding with metals in deprotonated form. The presence of new band ν(C-O$^-$) appears at 1270 cm^{-1} also confirms it. There are three CO group in the ligand, in which two amide ν(CO) bands occurs at same frequency or slightly shifted. It indicates that amide CO are not involved in complexation. While CO band of glyoxal is shifted towards lower frequency by 15-20 cm^{-1} in all complexes. This confirms glyoxal CO is participated in complex formation[15]. The ν(CN) bands observed at 1642 cm^{-1} in the spectra of gssdh, shifted to a lower frequency by 12-18 cm^{-1} in their metal complexes suggesting coordination through the two azomethine groups[16]. N (N-N) band present in ligand also shifted by 30-33 cm^{-1} to higher frequency in all-metal complexes shows its involvement in complex formation. All the metal complexes have some new weak bands in the range 520-540 cm^{-1} for ν(M-O) and 470-485 cm^{-1} for ν(M-N) band, as shown in (Table 2).

^{1}H and ^{13}C NMR

The ^{1}H NMR spectra of ligand possess multiplet signals due to aromatic protons at δ 6.70-7.35 (4H), two NH protons as a singlet at 7.57 (1H) and 7.68 (1H), two CH=N protons as a singlet at 5.55 (1H) and a doublet at 5.8 (1H, J = 7.1 Hz), Ar-OH as singlet at 5.35 (1H) and HC=O proton as a singlet at 10.10 (1H) ppm.

In the spectra of [Zn(gssdh-H)]Cl, both -NH protons show a slightly downfifield shift as compare to gssdh, which is indicative of deshielding of -NH proton and in turn shows interaction of the two CH=N groups with the metal ion.

The signals for aromatic protons show a minor shift in their position in the complex. A significant downfield shift in HC=O signal is characteristic of the C=O group of glyoxal in bonding with the metal ion (Table 2). The gssdh exhibits signals for two CH_2 as a singlet at δ3.85 (4H); there is a minor shift in the position of δCH_2 and aromatic protons. The absence

Table 1 — Magnetic moments, electronic spectral bands of the complexes

Complex	μ_{eff} (B.M.)	Electronic spectral band in nujol		Electronic spectral band in DMSO solution	
		Band maxima(nm)	Assignments	Band maxima(nm)	Assignments
[Co(gssdh-H)]Cl	4.35	680, 378	${}^4A_2(F)\rightarrow{}^4T_1(P)$ CT	1065, 520, 436	${}^4T_{1g}(F)\rightarrow{}^4T_{2g}(F)$, ${}^4A_2g(F)$, ${}^4T_{1g}(P)$
[Ni(gssdh-H)]Cl	diamagnetic	818, 478, 354	${}^1A_{1g}\rightarrow{}^1B_{1g}$, ${}^1B_{2g}$ CT	836, 470, 345	${}^1A_{1g}\rightarrow{}^1B_{1g}$, ${}^1B_{2g}$ CT
[Cu(gssdh-H)]Cl	1.80	630, 465, 395	${}^2B_{1g}\rightarrow{}^2A_{1g,}$ ${}^2B_{2g}$, ${}^2E_g$	680	${}^2E_g\rightarrow{}^2T_{2g}$

Table 2 — IR spectral bands of ligand gssdh and their metal complexes

Compounds	ν(OH)	ν(NH)	ν(C=O)	ν(C=N)	ν(C-N)	ν(C-O$^-$)	ν(N-N)	ν(M-O)	ν(M-N)
gssdh	3380 b	3220 b	1667b	1642s	1366s		967w	-	-
[Co(gssdh-H)]Cl		3213b	1669b, 1650m	1650m	1364s	1271s	1002w	533m	474m
[Ni(gssdh-H)]Cl		3208b	1667b, 1647m	1647s	1361s	1270s	1003w	534w	474m
[Cu(gssdh-H)]Cl		3213b	1669b, 1651m	1613m	1364m	1273s	1003w	522w	472w
[Zn(gssdh-H)]Cl		3210b	1668b, 1649s	1616s	1362m	1270m	1001m	533w	479w

Table 3 — ^1H and ^{13}C NMR spectral data of ligand and their metal complex

Compounds	^1H NMR signals (ppm)					
	CH_2	Aromatic protons	NH	-CH=N	Ar-OH	HC=O
gssdh	3.85s	6.70-7.35m	7.57s, 7.68s	5.55s, 5.8d	5.3s	10.1s
[Zn(gssdh-H)]Cl	3.9s	6.72-7.80m	7.80s, 7.65s	5.6s, 5.9s	-	10.3s
	^{13}C NMR Signals (ppm)					
	$>CH_2$	Aromatic Carbon	C-OH	-CH=N	>C=O	HC=O
gssdh	41.014	118.67-120.20	147.33	157.3, 156.3	162.51, 162.92	168.89
[Zn(gssdh-H)]Cl	41.03	120.3-123.56	148.26	157.8, 157.9	162.6,163.3	169.32

Table 4 — Antifungal activity of the ligand and its complexes

Compound	% Inhibition of spore germination				
	Aspergillus species (mg/mL)				
	1.0	1.5	2.0	2.5	3.0
gssdh	26	40	50	53	59
[Co(gssdh-H)]Cl	50	59	70	75	80
[Ni(gssdh-H)]Cl	49	60	65	70	78
[Cu(gssdh-H)]Cl	56	70	75	80	85
Miconazole	70	75	80	85	90

Table 5 — Antibacterial activity of the ligand and its complexes

Compound	*E. Coli* (mg/mL)				
	MIC (mg/mL)	diameter of inhibition zone (mm)		% Activity Index	
		1.5	3.0	1.5	3.0
gssdh	0.7	2	3	12	14
[Co(gssdh-H)]Cl	0.5	9	13	50	54
[Ni(gssdh-H)]Cl	0.4	12	16	70	78
[Cu(gssdh-H)]Cl	0.3	13	18	76	80
Ampicillin	0.2	18	20	98	100

of the signal due to Ar-OH proton in Zn(II) complexes of the ligand provides evidence for the deprotonation of the phenolic OH group of ligand during complexation inferred from IR spectral data also. The disappearance of -NH, -CH=N, and Ar-OH protons in the ligand in their D_2O exchanged ^1H NMR spectra confirm their assignments.

In the Zn(II) complex of gssdh ligand, the bonding through phenolic oxygen has been inferred from the deshielding observed in C-O- carbon compared with the ligand. The deshielding indicates the ligand's bonding in their Zn(II) complex through two azomethine nitrogen and HC=O group of glyoxal observed in -CH=N and HC=O carbon (Table 3).

Antifungal activity

The experimental antifungal activity data (Table 4) indicate that the ligand and its complexes show considerable activity against *Aspergillus* sp., at 1.5 and 3.0 mg/mL concentration. Their activity generally increases with increasing the concentration of the compounds. DMSO control has shown a negligible activity as compared to metal complexes and ligand. Co(II), Ni(II), and Cu(II) complexes show better antifungal activity than the ligand.

Antibacterial activity

Most of the complexes show better antifungal activity than the standard miconazole against a number of pathogenic fungi. The antibacterial activity of these complexes has been evaluated against *E. coli* and *Clostridium* sp. which shows a average activity (Table 5).

Conclusion

The paper includes the synthesis of unsymmetrical tetra-dentate Schiff base, glyoxal-salicylaldehyde succinic acid dihydrazone. The Co(II), Ni(II), Cu(II), and Zn(II) complexes of the above ligand have been synthesized by complexation with the preformed ligand. The IR and NMR spectra show that the Ligand bond with the metal ion through C=O, a phenolate group, and two C=N groups. The ligand and their complexes show moderate pesticidal activity. It has been found that complexes show more antifungal and antibacterial activity than the ligand due to metal-ligand bonds[17].

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Conflict of interest

All authors declare no conflict of interest.

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