Synthesis ,charactrazion and biological studies of metalscomplexes with Schiff base from sulfadiazine and teraphthaldyhyde

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Abstract:

Some new complexes of Schiff base ligand derived from sulfadiazine and terephthaldyhyde with Cu(II),Ni(II) ,Fe(II) ions were synthesizedand characterized by UV-Vis spectral analysis, Fourier transform infrared spectroscopy (FT-IR), H^1 – NMR ,biological activity The UV-Visible spectrophotometric analysis revealed 1:2 (metal-ligand) stoichiometry for all complexes and assigned an octahedral geometry.while the appearance of ν(O-H) due to water moleculesin all complexes and the lowering shift of the stretching frequency of the ν(CH=N) azomethine band in the ligand after complexation, the schiff base ligand showed higher significant activity against one or more bacterial compared with complexes.

Keywords : Sulfadiazine , Schiff base ,metal complexes, biological activity.

INTRODUCTION

Schiff bases derived from amino and carbonyl compound are an important class of ligands that coordinate to metal ions via azomethine nitrogen and have been extensively. In azomethine derivatives, the -CH=N- linkages is essential for biological activity[1], several azomethine were reported to possess a remarkable antibacterial. antifungal, anticancer and diuretic activities ,Schiff bases have wide application in food chemistry[2] ,dye industry ,analytical chemistry,catalysis,fungicidal ,agrochemical and biological activities,sulfa drugs which were the synthetic antimicrobial agents from p-amino bezenessulphonamide[3] and are primarily bacteriostatic in action ,their antibacterial activity is thought to arise from similarity of their structure to the p-amino benzoic acid found in bacterial for folic acid synthesis[4],the sulpha drugs can be enhanced upon coordination with a suitable metal ion[5,6],schiff bases are among the most studied sulfa drug derivatives which have been used for numerous biological applications[7,8] .In this paper,we therefore ,present the synthesis , characterization and biological studies Cu(II),Fe(II), and

Ni(II)complexesofschiffbasedderivedfromsulfadiazineandteraphthaldehyde. .

EXPERIMENTAL

Chemicals

Sulfadiazine (Fluka) ,terephthalaldehyde (Fluka), CopperSulfate. phentahydrate ,Nickel sulfate.heptahydrate and Iron sulfate .heptahydrate(Fluka),Ethanol absolute ,DMF, andAcetic acid (BDH).

Instruments

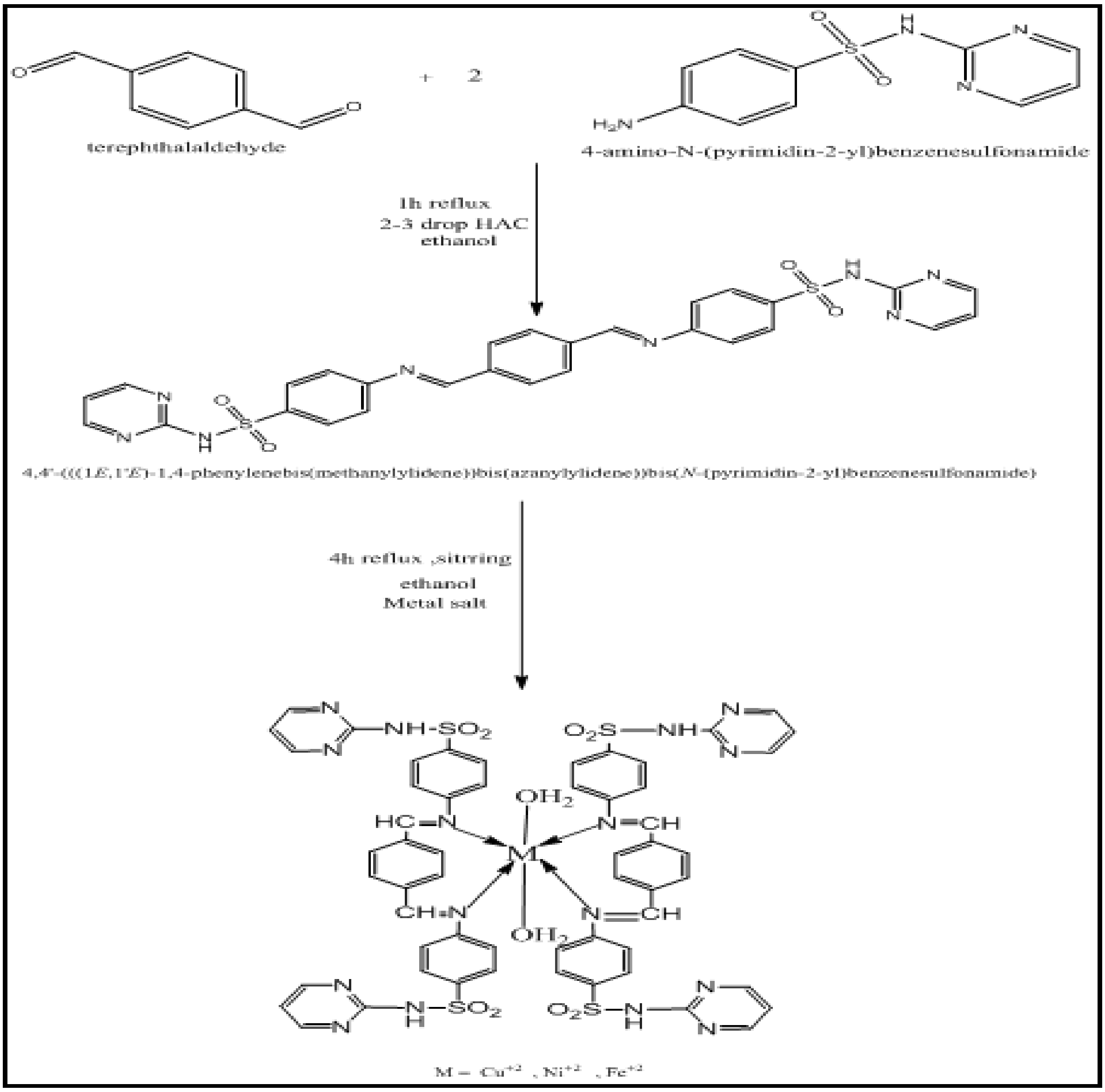
Melting points were recorded on Gallenkamp melting points apparatus without correction,IR Spectra were measured on Shimadzu 8400S spectrophotometer as KBr pellets in the region 4000-600cm^{-1},UV-Vis spectrometer cary(100)conc The ^{1}HNMR spectra were recorded in DMSO-d6 on Bruker 500MHz spectrometer using TMS an internal standard . Molar conductivities of 10^{-3} M solutions of the solid complexes in DMF were measured on the using Jenway 4010 conductivity meter,Magnetic measurements were measured by the Gouy method at roomtemperature using a magnetic susceptibility balance (Johnson Matthey alfa product, Model No.MKI).

SynthesisofSchiff basesligand(S_2T)

Schiff base was prepared by dissolving(0.5 g,2mmol)of sulfadiazine in 50ml absolute ethanol and(0.134g, 1mmol)terephthalaldehyde in 50ml absolute ethanol in 250ml round flask, the mixture was heated with reflux for 4hours ,during which the color of the solution changes to pale green . The formed pale green solid product was left to coagulate,then filtered off and was recrystallized from absolute ethanol .the yield was (88%),its melting point was 298C^{0}Scheme(1)..

General procedures for synthesis of the metal complexes

The following general procedures were used for preparing all of the complexes under investigation like that of Cu(II) complex: A solution of the $CuSO_4 .5H_2O$ (0.249 g ,2mmol) in 25ml absolute ethanol was added to solution of ligand (S_2T) (1.068 g , 1mmol) 25ml absolute ethanol with the molar ratio 2:1 (ligand : metal) the reaction mixture was heated to reflux for 24h .The color of solution was green in the beginning of reaction and then became pale green at the end the reaction , on cooling solid formed , the solid separated was filtered and washed with ethanol and then air-dried , the yield were between (62-84%)Scheme(1) .



Scheme(1) synthesis of schiff base ligand and metal complexes

Biological activity

Antibacterail activity was determined by using Mueller Hinton agar, Petri plates were prepared by pouring 10 ml of Mueller Hinton agar and allowed to solidify, Plates were dried and 100 µl of inoculum's suspension (approximately 106 cfu/ml of bacteria) was poured and uniformly, Whatman filter paper discs(5mm) were impregnated with 2 ml of ligand and complexes at concentration (10^{-3} M) and used to fill hole bored by cork borer in the inoculated agar. All the plates were incubated at 37 °C for 24 hours to obtain maximum growth in the culture media. For this concentration, the diameter of inhibition zone of growth minus the diameter of the disc was measured to estimate the degree of antibacterial activity ,by using DMF solvent[9,10].

Resultsanddiscussion

The schiff base of sulfadiazine ligand (S_2T) was prepared by the condensation of sulfadiazine with tetephthaladyhede in the molar ratio 2:1 ,the ligand (S_2T) was reacted withCu(II),Ni(II),Fe(II) ions to yield corresponding meta complexes (scheme 1),the physical data of ligand and corresponding transition metal complexes were listed in (table 1)..

Electronic spectra

Electronic spectral of the ligand (S_2T) were recorded in DMF ,and exhibited absorption bands at 254,298,310 nm, 39370,33557 3225cm^{-1} .The first band was correspond to transitions of the phenyl rings [11]. The second band correspond to the π → π*'s of the azomethine groups –CH=N– .The last band corresponds to the n → π* due to the non-bonding electrons on the oxygen and nitrogen atoms [12].The electronic of S_2T Cu complexes dissolved in DMF(Table 1). which exhibited shifttoward high wavelength and due to bands assigned toπ → π*,n → π* transition as well band due to^{2}Eg→$^{3}T_{2g}$ the electronic of S_2T Ni complex show absorption band at 26881cm^{-1} which due to$^{3}A_{2g}$ $^{3}T_{2g}$→while S_2T Fe complex exhibited band at 28169 cm^{-1}dueto$^{5}A_{1}g \rightarrow T_{2}g(G)$[13,14] .

Table (1) :The physical data obtained for the schiffbase and complexes

No	Fourmal of ligand and complexes	colour	M.P	Yield	$_{nm}$ λ	Electronic spectral data cm^{-1}	$_{m}\Lambda$Ohm^{-1} .m^{2}. mole^{-1}	$_{eff}\mu$ BM
1	$C_{28}H_{22}N_8O_4S_2$ S_2T	Pale green	298	88%	2982 54 310	39370,3355 7 32258	-	-
2	[Cu ($C_{56}H_{45}N_{16}O_{10}S_4$)($H_2O$)$_2$] S_2T Cu	Dark green	352	72%	254 380	39370 26315	1.32	1.99
3	[Ni ($C_{56}H_{45}N_{16}O_{10}S_4$)($H_2O$)$_2$] S_2T Ni	white	368	60%	254 372	39370 26881	2.5	2.3
4	[Fe ($C_{56}H_{45}N_{16}O_{10}S_4$)($H_2O$)$_2$] S_2T Fe	Yellow	386	63%	254 355	39370 28169	3.46	5.3

Infrared spectra

The vibrational modes of infrared spectrum of the ligand (S_2T)and metal complexes , assignments was listed in (Table 3) ,the N-H asymandN-H sym vibrations observed between (3259 -3105cm^{-1}) [15].the bands (-CH=N-) in the ligand (S_2T) due to 1651cm^{-1} while the lowering shift of the stretching frequencies of the bandoccurs at 1627-1626 cm^{-1} after complexation indicating the coordination of azomethine nitrogen atom to metal ions[16].The bandobserved at (1336-1153) of the (O=S=O). Anew band in the region (623cm^{-1}) due to (M-N) in all complexes ,the weak bands around (3584-3421cm^{-1}) indicate the presence of water molecules to complexes[17] .

Table (2) shows the famous IR frequencies of important bands of functional groups

compound	H_2O water	N-H sulpha	(C-H) str. Aromatic	(C-H) Alph	(HC=N) str. cm^{-}	C=N pyridine	SO_2 Asym. Sym.
S_2T	-	3259	3076 m	2937 as 2872 s	1651s	1577s	1323 1153
S_2T Cu	3421_w	3259	3039 m	2935 as 2875 s	1627s	1581s	1330 1157
S_2T Ni	3584_w	3259	3084 m	2931 as 2866 s	1627s	1581s	1334 1159
S_2T Fe	3430_w	3259	3041m	2941 as 2864 s	1626s	1577s	1336 1157

S=strong , w=weak , m= medium

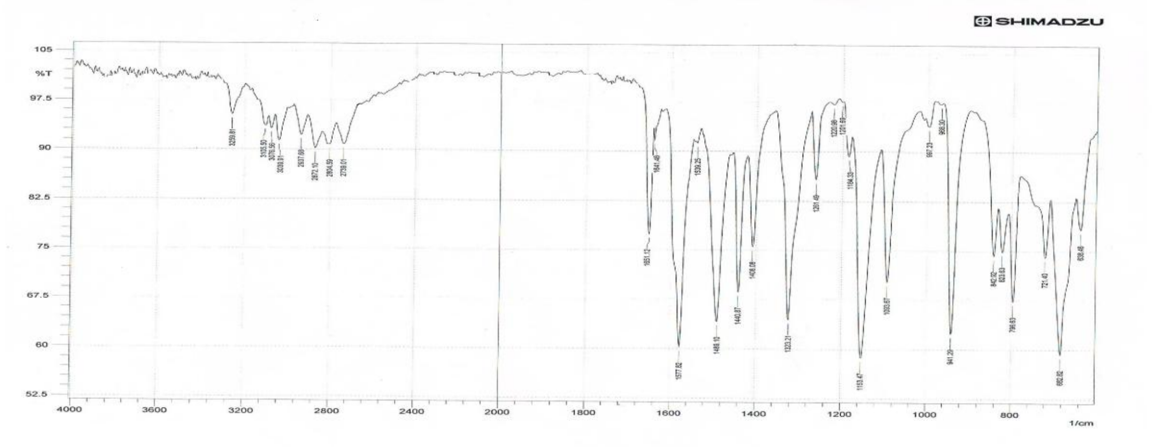


Figure 1: FT-IR Spectrum of ligand (S_2T)

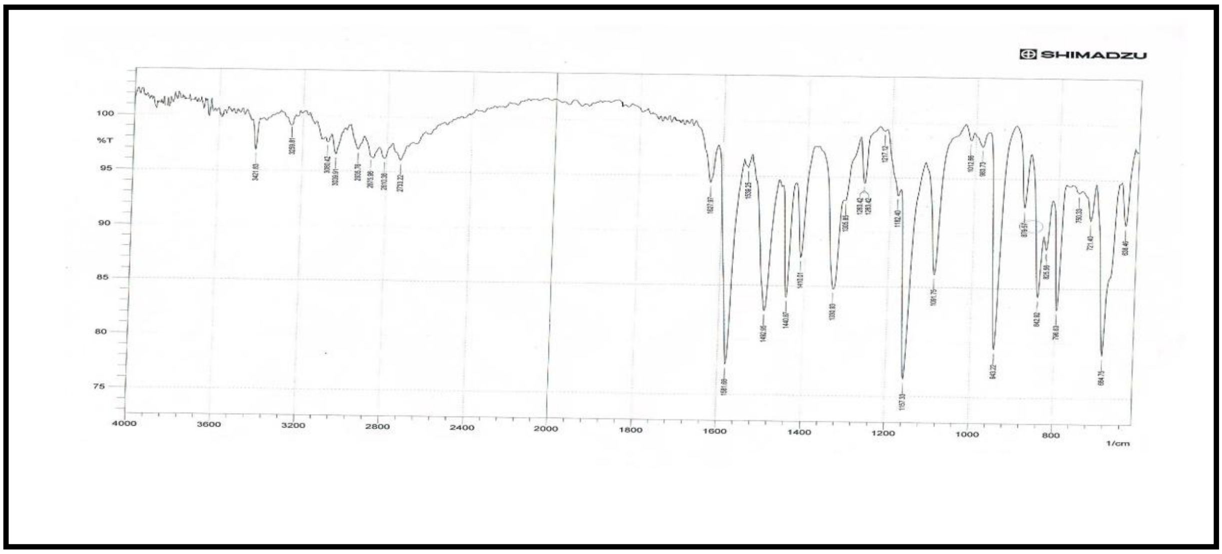


Figure 2: FT-IR Spectrum of complex(S_2T Cu)

Molar conductance

The observed molar conductance of all complexes in 10^{-3}M DMF solution at room temperature are listed in (Table 1) are foundwithin the range of 1.32-3.46 Ohm^{-1} .m^2. $mole^{-1}$ showing their non-electrolytic nature. The measured values of the magnetic moment μeff for complexes (Table 1), were 1.99, 2.3 and 5.3 B.M. respectively, indicating the octahedral structure [18]. The magnetic moment values measured were suggesting the possibility of the octahedral structures.

^{1}H-NMR spectra

^{1}H-NMR chemical shifts (ppm) of the ligand(S_2T) in DMSO-d_6 fig(3) the single observed at δ10.25ppm was due toNH proton of NHSO2 moiety[19] . While the signal at δ10.14 ppm attributed to NH proton ofwas assigned to the azomethine group (-CH= N-)[20] ,the aromatic protons in the region δ6.57 – 8 ppmandPyrmidine ring protons appear at δ8.08 – 8.76 ppm.

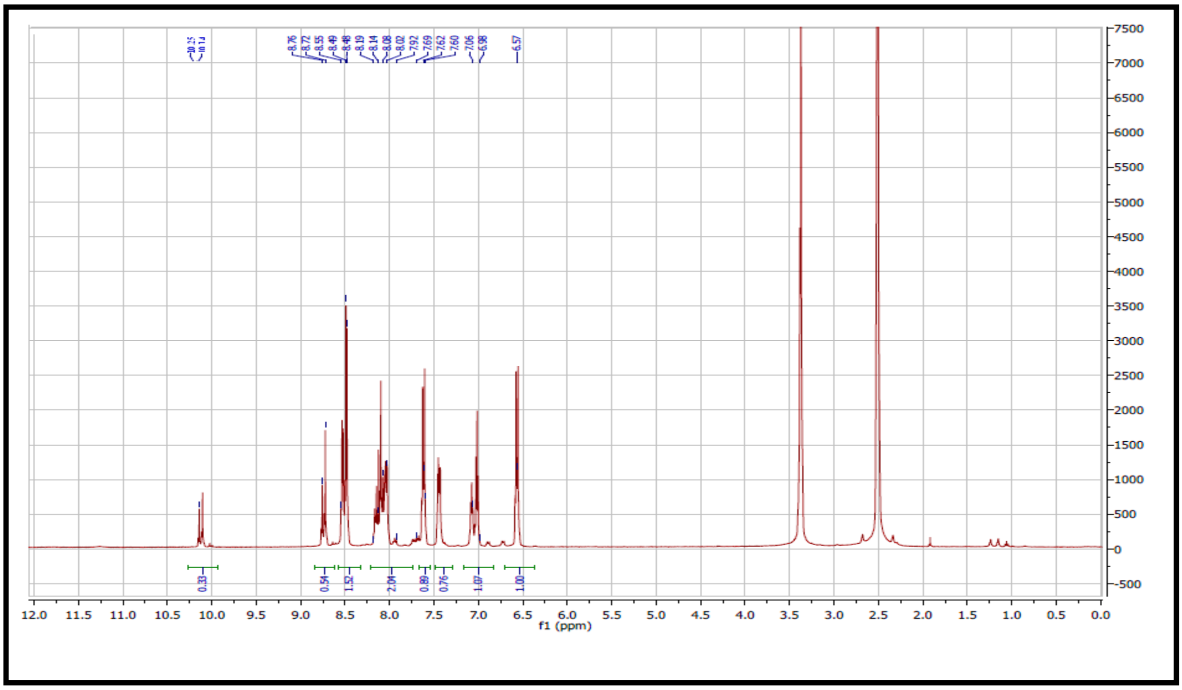


Figure 3: 1H-NMR Spectrum of ligand(S_2T)

Biological activity

Theantibacterial activity of ligand (S_2T) and complexes Cu(II),Ni(II),Fe(II),was determined against three bacterial stains which are listed in table (3) .The result show the complexes Cu(II),Ni(II),Fe(II) did not have any activity at concentration (10^{-3} M) compared with ligand the showed activity against Bacillus with azone inhibition (15 mm) than that of Staphyllococcus (10mm) and E.coli (12mm) at concentration (10^{-3} M)[21-23].

Table (3) : biological activity of ligand (S_2T)

Bacterial	Ligand (S_2T)	Diameter of inhibition zone (mm)
		(10^{-3} M)
Bacillus	1	15
Stapyllococcus	1	10
E.coli	1	12

Stapyllococcus

Bacillus

E.coli

Figure4: biological activity of ligand and complexes

Conclusions

The synthesized Schiff base ligand derived from sulfadiazine has been coordinated with the Cu(II), Ni(II) and Fe(II) metal ions through azomethine-N and water molecules and their structures have been confirmed by analytical and spectral data, IR^1 ,H-NMR spectra The magnetic moment. theresults proved that prepared complexes showed octahedral geometry and biological activity of ligandshowed higher to significant activity against one or more bacterial compared withcomplexes.

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