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SYNTHESIS AND CHARACTERIZATION OF IRON (III) COMPLEX CONTAINING SCHIFF BASES DERIVED FROM SULFADIAZINE

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ABSTRACT : Schiff base compound [(4-((2-hydroxynaphthalen-1-yl) methyleneamino)-N-(pyrimidin-2-yl)benzenesulfonamide] (L) was synthesized by reaction Sulfadiazine with 2-hydroxy naphthaldehyde in glacial acetic acid as solvent and catalyst. New complex (Lx) of Fe(III) with organic ligand Schiff base was also prepared. Ligand and complex were identified by elemental analysis, magnetic moment, ^{1}H-NMR spectrum and molar conductance along with electronic and infrared spectral analysis. Octahedral geometry around this metal ion has been proposed on the basis of magnetic and spectral studies.

Key words : Sulfadiazine, Schiff base, ligand and complex.

INTRODUCTION

Sulfadiazine [4-amino-N-2-pyrimidinylbenzene sulfonamide] (Lida Fotouhi *et al*, 2013) is one of sulfonamide group of antibiotics that using as a veterinary medicine (Chengshai *et al*, 2009). It eliminates type of bacteria which cause infections by preventing preparation of folic acid which bacteria was needed for growth. It is used for the treatment of many of infections for example toxoplasmosis, malaria, urinary tract infections and a certain type of brain infections (Ajibade *et al*, 2006). Sulfa drugs was possess (-SO_2NH-) Moiety, which is very important toxophoric functional group, conceder chemotherapeutically important such as sulfadiazine (silver sulfadiazine), sulfathiazole, sulfamerazine (NidaIqbal and Imran, 2009). Generally, there are two reactive position in sulfadiazine, the first sulfonamide group and the second aromatic amine (Zhaoliang *et al*, 2000). Schiff bases are the organic compounds containing azomethine group (HC=N-) synthesized by condensation of a primary amine with (aldehyde or ketone) an carbonyl compound (SofianSaleh *et al*, 2012). They were first reported by Schiff in 1864 (Ashraf *et al*, 2011) and the formula of the Schiff base is (RR'C=NR'') where, R, R', R'' is an alkyl group or phenyl group that makes Schiff bases a stable imine (Henry and Lange, 1977). Schiff bases are the important organic compounds showed wide spectrum of biological activities and many applications (Li *et al*, 2003). The common oxidation states shown by iron are +2 and +3. Almost all the aqueous chemistry of iron is confined to the +2 and +3 oxidation states. Fe (III) ions form a large number of complexes than Fe(II) ion. In most of these, the coordination number of Fe(III) ion is six and the complexes formed are octahedral (Cotton and Wilkinson, 1999). In view of these factors, in this research synthesized new complex of Fe(III) ions with linked Schiff bases containing oxygen and nitrogen donor atoms.

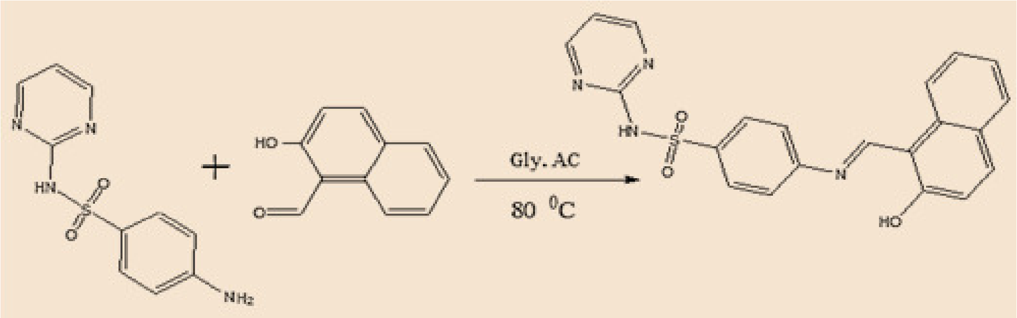
MATERIALS AND METHODS

All chemicals and solvents were highest purity obtained from Fluka, The Merck and BDH. The melting points were determined on a Electrothermal, melting point 9300. Elemental analysis (C.H.N) were obtained using Euro VECTOR (C.H.N) elemental analyzer. IR spectra were recorded using KBr discs in the range (4000-400) cm^{-1} on FTIR Test scan Shimaduz model 8300, while the (UV-Vis). Spectra recorded in ethanol on Shimaduz model 1650PC. Molar conductance measurements were carried out at lab. temperature in DMF (10-3 M), HANNA model 214EC conductivity meter. The pH measurements by using pH-meter Hanna. The metal content of the complex was determined by using atomic absorption technique by Perkin-Elmer model 2280.

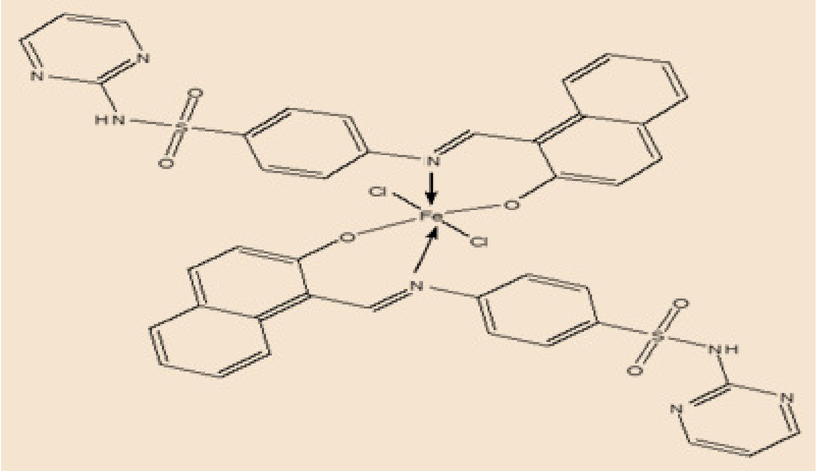
Preparation of ligands

(4mmol, 1gm) Sulfadiazine dissolved in (20 ml) glacial acetic acid and 2-hydroxy-1-naphthaldehyde (4mmol, 0.688 gm) was added drop wise. The resulting mixture was stirred at (80 ^{0}C) for (14) hr. Then, the TLC showed that the reaction was completed by using (hexane: ethylacetate, 4:1). The solution was separated by using diethyl ether. The organic layers were dried over $MgSO_4$. Removal of the solvent and recrystallize.

- (4-((2-hydroxynaphthalen-1-yl) methyleneamino)-N-(pyrimidin-2-yl) benzene sulfonamide (L)



Scheme 1 : Preparation of Ligand.



Scheme 2 : Structure of Fe(III) complex with Ligand.

- Chemical Formula: $C_{21}H_{16}N_4O_3S$
- Elemental Analysis:
- Calculate: H, 3.99; S, 7.93; C, 62.36; N, 13.85
- Found: H, 3.89; S, 7.873; C, 62.144; N, 13.778
- m.p. 240-242 , yield 60%
- **IR** (KBr disc, cm^{-1}): 3340 (N-H sulfonamide Str), 3070 (Aryl C-H),2944,2843 (Alkyl C-H), 1661 (C=O str), 1537 (C=N str), 1312 (SO_2 sulfonamide sym.) 1147 (SO_2Asym.str.), 1425-1600 (Aromatic ring).
- **^{1}HNMR** (ä ppm), (DMSO-*d6*) : 11.321 (SO_2-NH), 8.724 (HC=N pyrimidine ring) , 4.003 (C-Htriazoline ring), 8.615-6.625 (Aryl C-H),3.19 (N-$(CH_3)_2$.

Preparation of metal complex

To a hot solution of ligand (2 mmol) in 30mL methanol, the ferric chloride (1 mmol) in methanol was added drop wise. The pH of the solution was adjusted to 7 to 8.5 by adding alcoholic ammonia. The contents were digested for 4 h. The Fe (III) complexes were filtered in hot condition, product washed several time with hot methanol and then by petroleum ether and dried in vacuum.

- Chemical Formula: $[Fe(C_{42}H_{30}Cl_2N_8O_6S_2)]$
- Elemental Analysis:
- Calculate: C, 54.03; H, 3.24; N, 12.00; S, 6.87; Fe, 5.98
- Found: C, 53.889; H, 3.109;N, 11.977; S, 6.721; Fe, 5.78

The structure of metal complexes is shown Scheme 2.

RESULTS AND DISCUSSION

Ligand derived from reaction of Sulfadiazine with 2-hydroxy naphthaldehyde in glacial acetic acid. The [C.H.N.S] analysis of synthesized ligand was accepted agreement with the calculated percentage of elements. ^{1}H-NMR spectrum consider good evidence for prepared ligand (Fig. 1). The complex is colored solid and stable to air. It is insoluble in methanol, ethanol and water but soluble in DMF, DMSO. The synthesized ligand (L) was characterized by ultra-violate-visible (UV-Vis) spectroscopic technique and recorded in ethanol on Shimadzu model 1650P. The spectrum of the ligand in ethanolic solution (10^{-3} M) gave mainly three peaks, at (323 nm) and (377 nm due to the moderate energy ð-ð* transition of the phenyl rings. At the (487 nm) peak (ë max) due to the ð-ð* transition which take place from benzene ring by intermolecular charge- transfer through

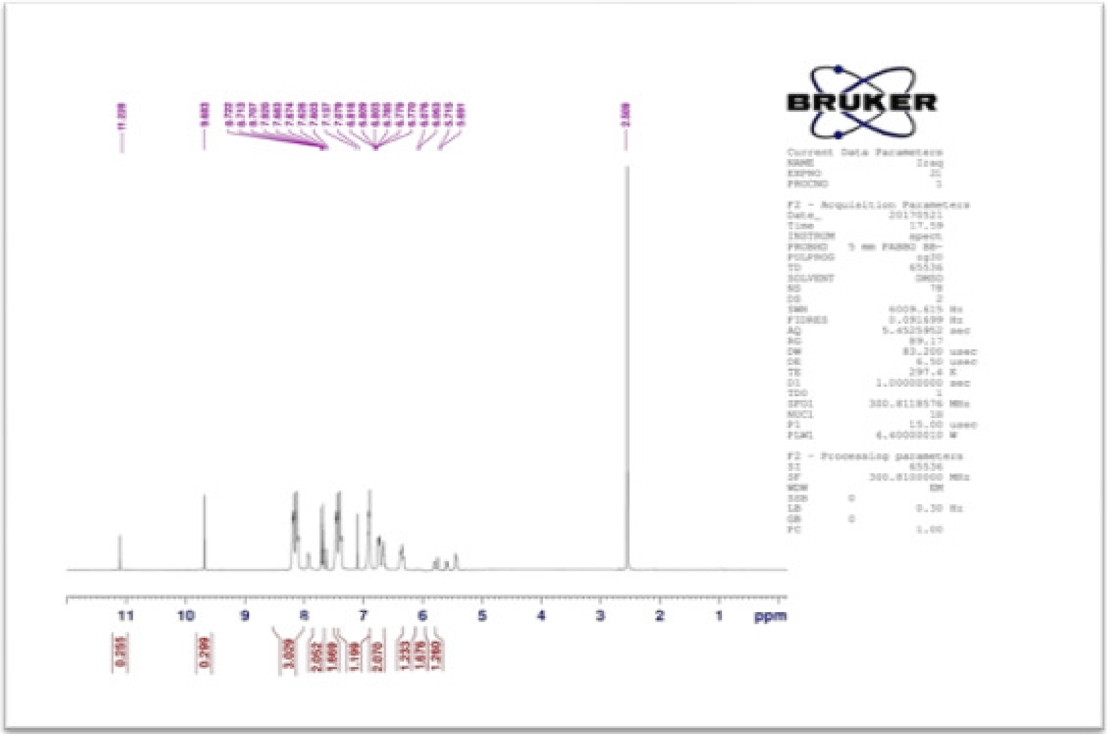


Fig. 1 : ^{1}HNMR Spectrum of ligand.

Table 1 : Antibacterial activities of compounds (L and LX).

Comp. No.	***Escherichia coli***		***Staphylococcus aureus***		***Pseudomonas aeruginosa***	
	300 ppm Zone of inhibition (mm)	**600 ppm Zone of inhibition (mm)**	**300 ppm Zone of inhibition (mm)**	**600 ppm Zone of inhibition (mm)**	**300 ppm Zone of inhibition (mm)**	**600 ppm Zone of inhibition (mm)**
L	30	50	40	60	35	60
LX	40	70	45	70	50	80

the azomethine group (Schiff base) (-C=N). Interaction of the (Fe III) ion with the ligand (L) has been studied in solution; An aqueous ethanolic solutions were always performed over wide molar concentration and acidity range. The interaction of the iron ion (Fe metal) with the ligand manifest itself in the absorption spectra by the appearance of a peak in the range (511 nm). A great bathochromic shift obtained in the visible region was determined in the complex solutions spectra with respect to that of the ligand alone. The high shift in the (ëmax) gave a good indication for complex formation. The Infrared spectra of the metal complexes were recorded between 4000-400 cm^{-1}. In IR spectra of free ligands, the band observed in the range 1622-1562 cm^{-1} were assigned to the C=N stretching frequencies, 3326 cm^{-1} (N-H) stretching of sulfonamide, 3410 cm^{-1}(OH) stretching, 3070 cm^{-1} (Aryl C-H) stretching, 1340 cm^{-1} (SO_2 sulfonamide sym.), 1122 cm^{-1} (SO_2Asym. Stretching. The good evidence of bonding is revealed by the appearance of band at 477-530 cm^{-1} (Fe-O), 432- 478 cm^{-1}(Fe-N) (Anil *et al*, 2014). The molar conductance values of all the complex in DMF at a concentration of 1X10^{-4} M fall in the range 25.44 mho.cm.^{2}mol.$^{-1}$. The conductance value is low molar indicates non-electrolytic nature of complex (Ramachandra *et al*, 2009). The Fe (III) complex show magnetic moment values are in the range 3,22 B.M. at room temperature suggesting high spins octahedral geometry (Abbas *et al*, 2013).

Applications

Schiff bases (L) and their Fe (III) complex (lx) was screened (at 300 and 600 ppm) *in vitro* for their antibacterial activity against of types of bacteria such as (*Sta. aureus*, *E. coli* and *Pse. aeruginosa*). The ligand showed 30-60% inhibition (Table 1). The complex had greater antibacterial activity than the corresponding ligand 40-80% inhibition.

CONCLUSION

In the present study, reported the synthesis of Fe(III) complex of azomethine (Schiff base) ligand derived from reaction of Sulfadiazine with 2-hydroxy naphthaLdehyed in glacial acetic acid. Infrared spectral studies suggest the metal-ligand coordination at azomethine and OH group. Further, by elemental analysis, molar conductivity measurements, magnetic susceptibilities, electronic absorption, structure for these Fe(III) complex is assigned as high spin octahedral structure.

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