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Preparation and Characterization of Mn(II), Co(II) and Cu(II) Complexes with Schiff Base Derived from Sulfadiazine and 2-Acetylfuran

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ABSTRACT

Three new complexes of Mn(II),Co(II) and Cu(II) with Schiff base derived from condensation reaction between sulfadiazine and 2-acetylfuran in methanol were synthesized and characterized based on solubility screening, melting point/decomposition temperature, Infrared spectroscopy elemental analyses and molar conductance measurements. The Schiff base which was isolated as a yellow powder, has a melting point temperature of 209^{o}C and percentage yield of 89.40%. The Schiff base and its metal (II) complexes were soluble in polar organic solvents and insoluble in non-polar solvents. The values of the decomposition temperature for the complexes were high in the range of 217-243 ^{o}C. Appearance of a new band at 1618 cm^{-1} in the IR spectrum of the Schiff base confirms the formation of azomethine linkage between sulfadiazine and 2-acetylfuran. Upon coordination to the metal ions the band for v(C=N) stretching undergoes a shift to higher wave numbers. The bands within 668– 687cm^{-1} attributable to v(M-N) vibration modes. The empirical formula calculation of the complex compounds indicated the formula [ML_2]. The molar conductance values of the complexes were low, which indicate the non-electrolytic nature of the metal complexes.

Keywords: Schiff base, Metal Complex, Sulfadiazine, 2-acetylfuran.

1.0 INTRODUCTION

Though the discovery and development of antibiotics are among the most successful achievements of modern science and technology for the control of infectious diseases, an increase in factors that bring about the diseases and an increase in number of multi drug resistance pathogens necessitate the search for new compounds with modified microbial activity. Modification of drug with transition metals helps to optimize the delivery of the drug to the active site. It is said to be better therapeutic agent (Chandra and Gupta, 2005) than their organic counterpart which are sometimes very selective in their activities and at the same time are very difficult to be extracted (Ming, 2003). Some of these drugs may have high toxicity (Zhang *et al.,* 2001).

Complexation of drug with metal ion is known to bring a drastic change in their biometric properties, therapeutic effects and pharmacological processes for instance Modification of drug with metal ion help to optimize the delivery of the drug to the active site (transition metals are suitably used because of their high thermodynamic stabilities). Schiff bases and their transition metal complexes have been reported to have an extensive application in biological science and material science due to their great flexibility. There are number of antibiotics and chemotherapeutics available for medical use but due to development of antibiotic resistance, there is need for a continue researches for the discovery of new compounds endowed with microbial activity that is clearly different from the well-known classes of antimicrobial agents to which many clinically pathogens are now resistant.

Schiff basesare organic compounds synthesized by the reaction between an aldehyde or a ketone with primary amines to form azomethine**-C=N-** (Zhong and Qin, 2014).They are type of ligand neutral or ionic molecules that binds to a central atom to form a co-ordination complex. The azomethine (imine; aldemines for aldehydes and ketoimines for ketones) is particularly used for binding metal ions (usually transition metals) via the N-atom lone pair, (Arulmuruganet *et al.*, 2010) especially in combination with one or more donor atoms to form polydentate chelates. (Munro, 2003). Schiff base are excellent chelating agents in the presence of a functional group like –OH or when –SH is present close to the azomethine group so as to form a five or six membered ring with the metal ion (Mishra *et al.,* 2013). Schiff bases are prepared by refluxing mixtures of amine and the carbonyl group in an organic solvent at different temperatures and reaction time but reaction with ketones are slower than that with aldehyde and higher temperatures and longer reaction time are often required (Jarrahpour and Khalili, 2006). Schiff bases and its transition metal complexes have an extensive application in material science and biological science example it is used as antibiotics, catalysts, and in spectrophotometric analysis (Agrawal*et al.,* 2013) as analytical reagent (Buhmann *et al.,* 1998), and also in the field of polymer-coating, ink, pigment (Whitesell, 1998), fluorescent materials (Tsai *et al.,* 2011) and catalytic reagent (Qiu *et al.,* 2001) due to their high sensitivity and selectivity.

Sulfonamides are well known antibacterial compound that has been in use for around 50years, they work by disrupting the production of folic acid that bacteria and human cells use for producing proteins. Its metal complexes drugs have drawn attention from different scientific community because of their superior clinical application (Sharfalddin *et al.,* 2021). They were the first effective chemotherapeutic agents employed for prevention and cure bacterial infection. Sulfonamide and azosulfonamide derivatives were known to be biologically versatile anticancer, antimalarial and antitubercular drugs. N-substituted sulfonamides are highly antibacterial and anti-fungal and are the most widely used antibacterial agent in the world because of their low cost, low toxicity and excellent activity against bacterial diseases, but due to the well known problems of its therapy, relating to the growing bacterial resistance, adverse effects and low bioavailability, sulfonamides are modified to Schiff bases and complexes that show available coordination mode depending on the reaction conditions and the complexes could be isolated and studied (Mansour, 2014).

This research work was aimed to synthesize Schiff base derived from sulfadiazine with 2-acetylfuran and some divalent metal complexes. The synthesized compounds will be analyzed and characterized using Infrared spectroscopy, elemental analysis, conductance measurements, melting points/decomposition temperatures, solubility test. The research will help in the discovery of new metal Schiff base complex compounds.

2.0 MATERIALS AND METHODS

All glass wares were well washed with detergent rinsed with distilled water and dried in an oven before use. All reagents and solvents used were of analytical grade (AnalaR) purchased from Aldrich and were used without further purification. All weighing was carried out on electric Metler balance model AB 54. Molar conductance was done on Cyberscan 500 model. Melting points and decomposition temperature were determined on Straut Scientific Melting point apparatus model SMP3.

2.1 Synthesis of the Schiff Base

An aqueous solution (15ml) of sulfadiazine (0.01 mol) was mixed with an ethanolic solution of 2-acetylfuran (0.01 mol/35ml) and the mixture was refluxed for 4h, where the color of the solution gradually changes. After that, the reaction mixture was acidified with few drops of dilute H_2SO_4 where a solid of sulfonamide Schiff-base was separated and re-crystallized from ethanol (Scheme 1) (Mansour, 2013).

SCHEME 1: Synthesis of the Schiff Base

2.2 Synthesis of the Metal(II) Complexes

The complexes were prepared by dissolving 2mmol of the ligand in 30ml ethanol, followed by dropwise addition of 10mlethanolic solutions of each of the metal salts (1mmol) (Elzahany *et al.*, 2008). The solution was mixed thoroughly in round bottom flask for 2 hours (Scheme 2). The filtrate was evaporated slowly at room temperature and the precipitate formed was dried in desiccator containing phosphorous pentaoxide.

SCHEME 2: Synthesis of the Metal (II) Complexes

3.0 RESULTS AND DISCUSSION

Condensation reactions between sulfadiazine and 2-acetylfuran in an eathanolic solution resulted in the formation of a yellow Schiff base with a percentage yield of 89.40% (Table 1). The Schiff base has a sharp melting point which indicates its purity. The reaction of ethanolic solutions of the Schiff base and the respective metal (II) chloride gave 79.85 – 89.89% of the metal (II) Schiff base complexes (Table 1). The metal (II) complexes have high decomposition temperature in the range of 217-243^{o}C which signifies thermal stability of the complexes. The Schiff base and its corresponding metal complexes were found soluble in polar solvents such as methanol, ethanol, DMSO, DMF etc, and insoluble in non-polar solvents such as benzene, diethylether etc. The empirical formula calculation of the complex compounds indicated the formula [ML2].

Table 1: Some Physical Data of FSDZ Schiff Base (FSDZ) and its Metal(II) Complexes

Compound	Colour	Melting Point (^{o}C)	Decomp. Temp. (^{o}C)	Percentage Yield (%)	% Elemental Analysis found (calc)			
					C	H	N	S
HL	Yellow	209	-	89.40	56.13 (55.90)	14.12 (14.35)	16.36 (16.15)	9.37 (9.41)
[MnL_2]	Brown	-	243	86.90	52.10 (51.93)	3.55 (3.81)	15.19 (15.01)	8.69 (8.71)
[CoL_2]	Pale Yellow	-	221	79.85	51.82 (51.90)	3.53 (3.49)	15.11 (15.07)	8.65 (8.62)
[CuL_2]	Dark Green	-	217	89.89	51.50 (51.43)	3.51 (3.57)	15.02 (15.09)	8.56 (8.60)

Key: $L^{-1} = C_{16}H_{13}O_3SN_4^{-1}$

The significant IR bands of the free Schiff base (FSDZ) and its corresponding metal complexes were used as a tool to detect the influence of a metal bonding on the ligand vibration in the complexes. In the FT-IR spectrum of Schiff base, the strong band observed at 1618cm^{-1} can be assigned to the azomethine (-HC=N) stretching vibration (Nakamoto, 2009). In the spectra of metal complexes, the -HC=N stretching vibration band is shifted to a higher wave number as presented in Table 2. This increase indicates the coordination of the azomethine nitrogen to the metal ion. Band observed at 1596 cm^{-1} in the spectra of the Schiff base was due to *v*(N-H) stretching vibrations (Nakamoto, 2009). A shift to higher wave number was observed in the spectra of the metal complexes in the range of 1577-1581 cm^{-1}. This shift suggests coordination between the metal ion and the nitrogen (form the sulfadiazine moiety). IR bands in the range of 668-687 cm^{-1} are due to metal nitrogen (M-N) bonds. These bands were absent in the spectra of the Schiff bases. This indicates that there is coordination between the metal and the lone pair of electron on the nitrogen atom of the ligand.

Table 2: Infrared Spectral Data of FSDZSchiff Base (FSDZ) and its Metal(II) Complexes

Compound	*v*(C=N) (cm^{-1})	*v*(N-H) (cm^{-1})	*v*(M-N) (cm^{-1})
HL	1618	1596	-
[MnL_2]	1652	1577	683
[CoL_2]	1652	1577	668
[CuL_2]	1652	1581	687

Key: $L^{-1} = C_{16}H_{13}O_3SN_4^{-1}$

Electrical behavior of the complexes was investigated in 10^{-3} M DMSO solution and the molar conductivity values for Mn(II), Co(II) and Cu(II) Schiff base complexes were found in the range of 19.00-35.10 ohm^{-1} cm^2 mol^{-1} as seen in Table 3. The molar conductivity values recorded for the metal complexes were low (Table 3), which indicates non electrolytic complexes (Ali *et al.*, 2013).

Table 3: Conductivity Measurements for Metal Complexes with FSDZ Schiff Base (FSDZ) in 10^{-3}M DMSO Solution.

Compounds	Concentration	Electrical Conductivity ($ohm^{-1}cm^{-1}$)	Molar conductivity ($ohm^{-1}cm^2mol^{-1}$)
[MnL_2]	$1x10^{-3}$	$35.10x10^{-6}$	35.10
[CoL_2]	$1x10^{-3}$	$19.36x10^{-6}$	19.364
[CuL_2]	$1x10^{-3}$	$19.00x10^{-6}$	19.00

Key: $L^{-1} = C_{16}H_{13}O_3SN_4^{-1}$

4.0 CONCLUSION

Sulfonamide Schiff base and its corresponding metal (II) complexes with Co(II), Cu(II) and Mn(II) have been synthesized and characterized via IR spectroscopy, C H N S microanalysis, solubility test, melting points, decomposition temperature and conductivity measurements. Analysis of the experimental data suggests that the Schiff base coordinate the metal ion in a bidentate manner through N-atom of the sulfadiazine and the N-atom of the azomethine, thus act as a bidentate ligand. The analytical data showed that the metal-ligand stoichiometry in the complexes is 1:2.

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