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Solvent-Free and Solution based: Synthesis and Biological Evaluation of Fe (II) Complex Derived from Aspirin

S. Jibril[1*]., H. Jibrin[2]., B. Isah[3]., and S. Abubakar[1].
[1](Department of Chemistry, AminuSaleh College of Education, Azare, Bauchi State, Nigeria)
[2](Department of Psychology, AminuSaleh College of Education, Azare, Bauchi State, Nigeria)
[3](Department of Educational services, AminuSaleh College of Education, Azare, Bauchi State, Nigeria)

Abstract
The new metal (II) complex [Fe (asp)$_2$ Cl$_2$] has been synthesized by both the solution and solid state methods and were characterized by elemental analysis, molar conductance, magnetic moment and spectral studies as well as biological evaluation. On the basis of spectral studies, magnetic moment and molar conductance, octahedral geometry was assigned to the complexes and suggest the free ligand as bidentate. The authors recommended the use of solvent-free synthesis over solution based due to its time management, inexpensive and non-hazardous.

Key words: *Solvent-free, solution-based, biological, complex*

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I. INTRODUCTION

Metals are essential cellular components that functions in several indispensable biochemical processes living organisms and transition metal complexes are very important in material synthesis and in biological system [1].While metals deficiency in the body can leads to so many diseases such as anaemia from iron deficiency, growth retardation from zinc deficiency and brain diseases for infants and heart diseases for adult from copper defiency[2]. Medicinal inorganic chemistry has been used in the exploitation of the properties of metals and their salts for the design of new drugs [3]. With the recent advancement of coordination compound complexes, the role of metals in medicinal inorganic chemistry to the design of innovative metal-drug and their application has been highlighted by researchers

Herein, we report the synthesis of Fe (II) complex derived from aspirin by both solvent-free and solution based synthesis, and also to evaluate the biological activity of the complex.

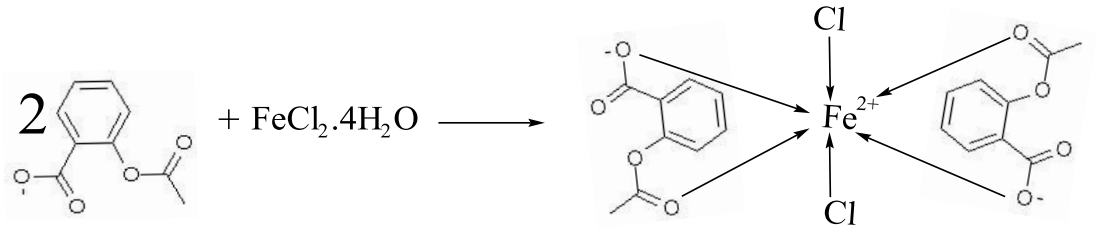
II. MATERIALS AND METHODS

Materials

Metal salts and active pharmaceutical ingredient of aspirin were purchased from sigma Aldrich and the entire chemicals were used without further purification. All the glass wares, mortar and pestle used was obtained from chemistry laboratory AminuSaleh College of Education, Azare, and washed thoroughly after each refluxing and grinding. IR and UV/Vis spectra were recorded using FTIR spectrometer of Agilent technology in the range of 400-4000 cm^{-1} and parking palmer spectrometer lambda 35 within the range of 200-700 nm both at Bayero University Kano. Molar conductivity was recorded using conductivity meter of model DD-307, melting point was carried out using Stuart melting point model ISMP 101 both at Bauchi State University Gadau. Antimicrobial test was carried out at the department of microbiology at Bayero University Kano.

Synthesis of the Complexes

A solution of aspirin (2mmol of aspirin in 10ml of distilled water); 1mmol of $NaHCO_3$ was dissolved in 10ml of distilled water and was added to the solution of aspirin drop wise with a constant stirring till clear solution was obtained, then 0.5g of $FeCl_2.4H_2O$ was added directly to a solution of aspirin with stirring. The solution was reflux for about 30minutes and brown precipitate was formed on cooling, then the solution was filtered and dry to give complex of Fe (II) [4]. The solvent-free synthesis was carried out by grinding 2mmol of aspirin and 1mmol of $FeCl_2.4H_2O$ using glass mortar and pestle for about 15minutes. The brown moist solid powder was obtained and recrystallized with water and filtered to give a clear crystal [5].



Scheme 1

III. RESULT AND DISCUSSION

The physical characteristics of the Fe (II) complex by methods of solvent-free and solution based synthesis were given in Table 1. From the results it shows that all the complexes are stable due to their higher values of decomposing temperatures of 175^0C and 188^0C tan free ligand of 160^0C and the conductance values of $27.3\Omega^{-1}cm^2mol^{-1}$ and 16.0 $\Omega^{-1}cm^2mol^{-1}$ that indicate the complexes to be non-electrolyte that falls within the solubility limit in 0.001ml in DMF which agreed with the proposed structure of the complexes [6].

Table 1: ***The physical properties of aspirin and its metal (II) complexes***

Ligand/ Compound	Methods	Colour	Melting Temperature (^{o}C)	Decomposition Temperature (^{o}C)	Conductance ($\Omega^{-1}cm^2mol^{-1}$)
Aspirin	-	White	160	-	-
$[Fe(asp)_2Cl_2]$	Solution based	Spine brown	-	175	27.3
$[Fe(asp)_2Cl_2]$	Solid state	Spine brown	-	188	16.0

Table 2: ***The Elemental analysis data of the complexes***

Compounds	Molecular formula (Molar Mass)	Methods	Microanalysis: found (calculated) % C	H	M
$[Fe(asp)_2Cl_2]$	$C_{18}H_{16}O_8FeCl_2$ (487.2)	Solution synthesis	44.87 (44.33)	3.43 (3.28)	11.43 (11.49)
$[Fe(asp)_2Cl_2]$	$C_{18}H_{16}O_8FeCl_2$ (487.2)	Solid state synthesis	43.69 (44.33)	3.45 (3.28)	12.08 (11.49)

The analysis data revealed that the C, H and Metal of the complexes synthesized by both methods are in agreement with the values found in theoretical data [7].

Table 3: ***IR spectral data and coordination mode***

Ligand/ Complexes	Methods	υ (C-O) cm^{-1}	υ (O - H) cm^{-1}	Carbonyl of carboxylate (C=O) cm^{-1}	Carbonyl of ester (C=O) cm^{-1}	M-O	M-Cl
Aspirin	-	1254-26	3372.69	1752.46	1581.71	-	-
$[Fe(asp)_2Cl_2]$	Solution based	1218.48	3238.93	1756.67	1625.72	426.60	758.34
$[Fe(asp)_2Cl_2]$	Solid state	1223.50	3234.72	1756.63	1625.66	426.00	705.45

The IR spectra for both the ligand and metal complexes are shown in Table 3 where the ligand behave as bidentate coordinated via one of the oxygen atoms of carboxylic acid with the displacement of hydrogen atom at $1756.63cm^{-1}$ and $1756.63cm^{-1}$ by both methods and oxygen atom of ester at $1625.72cm^{-1}$ and $1625.66cm^{-1}$, both through their carbonyl groups. The bands at $426.60cm^{-1}$ and $758.34cm^{-1}$, $426.00cm^{-1}$ and $705.45cm^{-1}$ by both methods which are absent in the free ligand are assigned to M-O and M-Cl stretching frequencies [8]. There are present of bands in the free ligand that were assigned to υ (C-O) cm^{-1} and υ (O-H) cm^{-1} stretching vibration which are shifted to a lower or higher frequencies in both the complexes which also, provide the evidence of coordination [9].

Table 4: ***Electronic spectra, magnetic moment and suggested geometry***

Ligand/ Complexes	Methods	Electronic spectra			Magnetic moment (BM)	Suggested geometry
		Wavelength (nm)	Energy (cm^{-1})	Transition		
Aspirin	-	209	47847	n- π^*		-
		227	44051	π- π^*		
		332	43103	C T		
$[Fe(asp)_2Cl_2]$	Solution based	230	43478	MLCT	5.67	Octahedral (oh)
		286	34965	$^5T_{2g}$-$^5T_{2g}$		
		336	29762	$^5T_{2g}$- E^2_g		
$[Fe(asp)_2Cl_2]$	Solid state	222	45045	n- π^*	5.55	Octahedral (oh)
		239	41841	π - π^*		
		555	39216	MLCT		
		294	34014	$^5T_{2g}$-$^5T_{2g}$		
		328	30488	$^5T_{2g}$ - E^2_g		

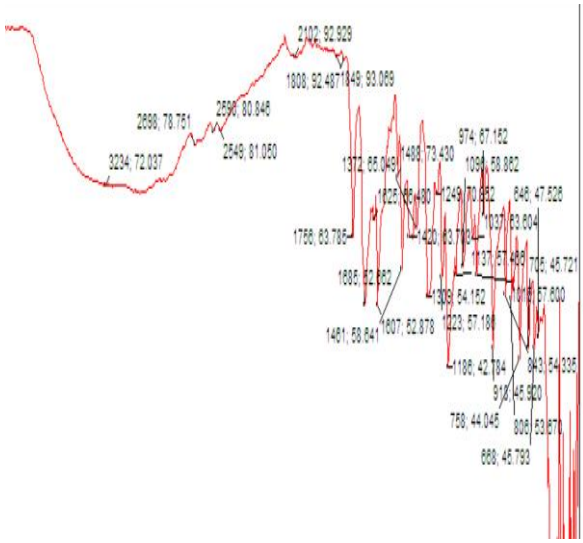


Fig-1: IR spectra of Fe (II) complex

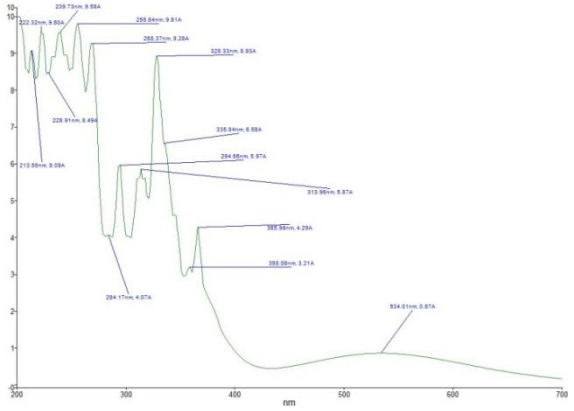


Fig-2: Electronic spectra of Fe (II) complex

The results for both electronic spectral data and magnetic moment values are presented in Table 4. The electronic spectra of free ligand showed three bands at 209nm, 227nm and 232nm which were attributed to $n-\pi^*$, $\pi-\pi^*$ and CT respectively [10]. The UV/Vis spectra of Fe (II) complex is presented in Figure 2. The bands appear at higher frequencies in both the Fe (II) complexes which falls within the suggested octahedral geometry [11] and magnetic moment values indicate the high spin octahedral complex which falls within the range of 5.5BM and 5.67BM by both methods [12].

Table 5: ***Anti-bacterial activity test of aspirin and its metal (II) complexes by both methods***

Ligand/ Complexes	Methods	Concentration (µg/ agar-well)	S. Aureus (mm)	E. Coli (mm)
Aspirin	-	4000	15	13
		2000	17	9
		1000	10	7
		500	8	-
$[Fe(asp)_2Cl_2]$	Solution based	4000	12	14
		2000	11	10
		1000	09	08
		500	08	-
$[Fe(asp)_2Cl_2]$	Solid state	4000	13	12
		2000	10	09
		1000	08	07
		50	-	-

Biological evaluation result shows that the free ligand (aspirin) is active against all the bacteria isolates at all concentration as presented in Table 5. $[Fe(asp)_2Cl_2]$ synthesized by solution based is active against all the

bacteria isolates at all concentration except E. Coli which is inactive at 500 µg[13]. While [Fe $(asp)_2$ Cl_2] synthesized using solid state method is active against all the bacteria isolates with the exception of S. Aureus and E. Coli which is inactive at lower concentration of 500 µg[14].

IV. CONCLUSION

The complex [Fe $(asp)_2$ Cl_2] has been synthesized using both solution based and solvent-free synthesis in molar ratio of 2:1 ligand to metal. The complexes were characterized using spectral studies, conductivity measurement, magnetic moment and elemental analysis. The electronic spectra and magnetic moment studies suggest the complexes as octahedral geometry and IR spectral studies provide the mode of coordination of all the complexes via carbonyl of carboxylate and ester[15]. Molar conductance rendered the complexes as non-electrolytes in DMF solvent.

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