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Liquid-Assisted Grinding: Preparation of 3d Complex Derived from Cloxacillin

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ABSTARCT: *3d complex derived from cloxacillin was prepared using liquid assisted grinding with water as a best possible green solvent. Elemental analysis data shows the complex to be represented as [Fe (clox)$_2$ Cl$_2$]. Spectral studies revealed the ligand as bidentate which coordinated to the metal ion through carbonyl group and also, both the spectral studies, magnetic moment value and conductivity measurement proposed the complex as octahedral geometry and rendered it non-electrolyte in nature in DMSO solvent.*

KEY WORDS: Liquid assisted grinding, preparation, complex, cloxacillin

INTRODUCTION

Pharmaceutical industries are making effort to protect people by reducing environmental impact from the exposure to pharmaceutical synthesis generated waste, the synthesis of active pharmaceutical ingredients required sustainable and environmental friendly synthesis. Recently, solid state synthesis attracts more attention to research communities, because of its cost effective, more convenient to use due to its unhazardous nature and also it reduce environmental contamination [1]. LAG (liquid assisted grinding) is one of the main types of solid state reactions in which a small amount of solvent (usually in drops) is used as an additive to enhance or control reactivity [2]. Therefore, the main aim of this research was to use the best possible green solvent water as LAG for the preparation of 3d complex of Fe (II) derived from cloxacillin and to examine its biological activity.

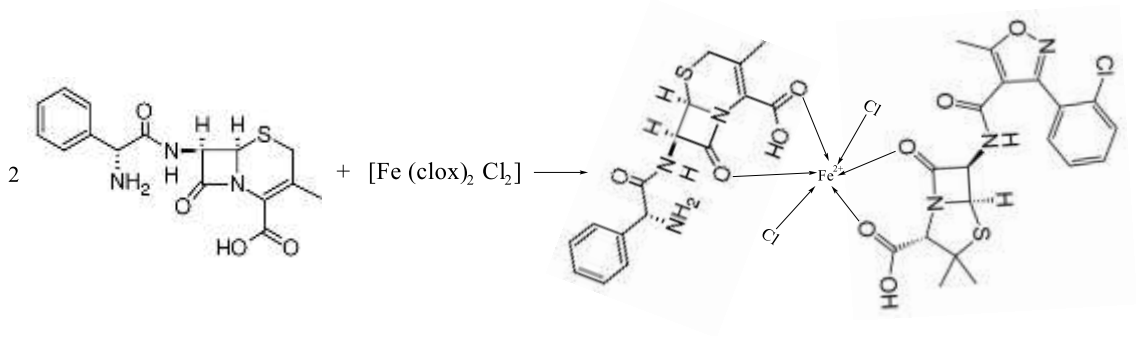
EXPERIMENTAL

Materials

All the reagents and APIs of cloxacillin were purchased from Sigma Aldrich and were used without any further purification.

Synthesis of the Complex

1mmol of $FeCl_2.4H_2O$ and 2mmol of cloxacillin were grounded with two drops of distilled water in a glass mortar in 1:2 ratios for 10-15 minutes. The deep brown product obtained was dried in dessicator [2].



Scheme 1

Apparatus and Methods used

Molar conductance measurement was done using DDS-307 conductivity meter using DMSO solvent at department of chemistry Bauchi state University Gadau.

PerkinElmer Lambda 35 UV-Visible spectrophotometer of 200-700nm wavelength was used for UV-absorbance measurement and IR spectral data by Agilent technologies in the range of 400 to 4000cm^{-1} both the results were recorded at the central laboratory Bayero University Kano.Magnetic susceptibility measurement of the complex was recorded using magnetic susceptibility balance of Sherwood scientific Cambridge UK.

RESULT AND DISCUSSION

The physical properties of both the ligand and the complex are presented in Table 1. Molar conductivity value in DMSO solvent ($1x10^{-3}$ ml) for the complex of Fe (II) is 14 $\Omega^{-1}cm^2mol^{-1}$ which falls within the range of non-electrolyte and it has been used to determine the geometry of the complexes [3].

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

Ligand/ Compound	Colour	Melting Temperature (°C)	Decomposition Temperature (°C)	Conductance ($\Omega^{-1}cm^2mol^{-1}$)
Cloxacillin	White	150	-	-
[Fe (clox)$_2$ Cl$_2$]	Deep Brown	-	221	14.0

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

Compounds	Molecular formula (Molar Mass)	*Elemental analysis*: found (calculated) %			
		C	H	N	M
[Fe (clox)$_2$ Cl$_2$]	$C_{19}H_{18}Cl_3N_3O_5SFe$ (491.9)	45.52 (46.35)	3.39 (3.66)	8.48 (8.54)	10.77 (11.38)

The elemental analysis data of the complex (C, H, N and M) shows the Fe (II) complex of cloxacillin to be represented as [Fe (clox)$_2$ Cl$_2$] [4].

Table 3: ***Electronic spectra, magnetic moment and suggested geometry***

Ligand/ Complexes	Electronic spectra			Magnetic moment (BM)	Suggested geometry
	Wavelength (nm)	Energy (cm^{-1})	Transition		
Cloxacillin	207	48309	n- π^*		-
	208	4955	π- π^*		
	238	42735	C T		
[Fe (asp)$_2$ Cl$_2$]	217	46089	n- π^*	5.92	Octahedral (oh)
	209	47846	π - π^*		
	335	29850	MLCT		
	351	28490	$^5T_{2g}$-$^5T_{2g}$		
	369	27100	$^5T_{2g}$ - $^5E^2_g$		

Electronic spectral data for both the ligand and the complex are presented in Table 3 in which the ligand shows three absorption bands at 207nm, 208nm and 238nm which were assigned to n-π^*, $\pi - \pi^*$ and CT band [5]. These bands also appear in the complex but are shifted to higher values which prove the coordination through between the ligand and the metal ion [6].

Table: 4: ***The IR Spectra Data of Cloxacillin and its Metal (II) complexes***

Compounds	ν(N-H) cm^{-1}	ν(O-H) cm^{-1}	ν(C= O) cm^{-1} β-lactam	ν(C=O) cm^{-1} of (CO_2^-)	ν(C=N) cm^{-1}	M-O	M-Cl
Cloxacillin	3514.85	3677.85	1771.53	1689.66	1603	-	-
Fe(clox)$_2$Cl$_2$	2970.88	-	1767.92	1547.81	1603	486	735.82

Keys: υ=wave number, M = metal, clox = cloxacillin, O = oxygen

The result of IR spectra of the ligand and the complex are presented in Table 4. The bands at 1771.53 cm^{-1} and 1689.66 cm^{-1} are assigned to carbonyl of amide and carboxylate of carboxylic acid which shifted to 1767.92 cm^{-1} and 1547.81 cm^{-1} respectively which suggested the evidence of coordination through carbonyl groups [7]. The bands at 486.00 cm^{-1} and 735.82 cm^{-1} which could not be traced in the spectrum of free ligand is tentatively assigned to M-O and M-Cl [8]. The above IR results rendered the free ligand cloxallin to be bidentate during complexation with metal ion.

Table 5: ***Antibacterial activity test of cloxacillin and its metal (II) complexes.***

Compounds	Concentration(µg)	S. Aureus (nm)	E. coli (nm)
Cloxacillin	4000	17	14
	2000	15	11
	1000	12	8
	500	10	7
$[Fe(Clox)_2Cl_2$	4000	13	12
	2000	10	9
	1000	8	7
	500	-	-
Control: Ciprofloxacin	500	35	30

Key: S. aureus = Staphylococcus aureus E. coli = Escherichia coli

The antibacterial studies shows the complex are active against all the bacteria isolates at different concentration except S. Aureus and E. Coli at lower concentration of 500 µg [9]. The results are compared with the standard antibiotics of ciprofloxacin.

CONCLUSION

The structure of the complex has been confirmed from the spectral studies (IR and UV-Visible), magnetic moment and molar conductance [10]. IR spectra suggest the ligand to be bidentate which coordinated to the metal ion through ν(C= O) of amide and one of the oxygen atom of carboxylate group. Elemental analysis results shows the complex to be represented as [Fe $(clox)_2$ Cl_2] while molar conductance value rendered the complex as non-electrolyte in DMSO solvent.

RECOMMENDATION

The authors recommended the use of solvent-free synthesis as its environmental friendly, cost-effective, give higher yield and conducted within shorter period of time.

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