



Synthesis and Study of Thermal Behaviour of Cu^{2+} and Fe^{2+} Complexes Derived from N-N¹-Diphenylhydroxylamineethylacetate

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ABSTRACT

The Schiff base ligand N-N¹-diphenylhydroxylamineethylacetate (DHA) prepared by the reaction of 2-phenylglycinemethylesterhydrochloride and salicylaldehyde was used to precipitate the transition metal complexes of Cu^{2+} and Fe^{2+} . These procedures were achieved using the microwave assisted technique regulated at 100°C for 30 min. Characterization of the ligand and complexes was carried out on the basis of physical properties, FTIR, XRD, UV-vis, spectroscopy. Unit cell dimensions which were obtained from the XRD crystallography analysis agreed with the establishment of orthorhombic crystal structure. The spectroscopic studies showed bands assignable to the azomethine nitrogen coordinated to the metal ion. The thermal decomposition of the complexes indicates the loss of lattice water and decomposition of the ligands as key to the interpretation of successive weight loss.

Keywords: N-N¹-dephenylhydroxylamineethylacetate, Salicylaldehyde, Schiff base, Thermogravimetry

INTRODUCTION

The synthesis and study of complexes with Schiff base ligands has gained influence on account of their easy accessibility, structural variety and their wide spectrum of biological and industrial applications (Iorungwa *et al.*, 2020a). The development in the area of bioinorganic chemistry has provoked research in the area of Schiff base complexes as a number of these compounds have been investigated due to their interesting biological activities such as biological modeling applications, ability to reversibly bind oxygen, antibacterial and antifungal properties (Razieh *et al.* 2013; Rabia *et al.*, 2016). In many instances, Schiff base complexes have been more efficient compared to the free ligands. Schiff base offer a versatile and flexible series of ligands capable of binding with metal ions to form complexes with suitable properties for many applications (Razieh *et al.* 2013; Fei – Ran *et al.*, 2014; Suman *et al.*, 2015).

Thermogravimetric analysis of Schiff base complexes and ligands are useful tools as it provides information about the thermal stability and decides whether the water molecules if found are inside or outside the inner coordination sphere of the central metal ion, and also suggests the general scheme for the thermal decomposition of metal complexes (Abdel – Kader *et al.*, 2013; Basim *et al.*, 2019).

Even though many Schiff bases derived from salicylaldehyde and substituted

salicylaldehyde and amines have been reported (Joshi *et al.*, 2011; Chandraleke and Chandramohan, 2014). Literature work on complexes formed from the condensation reaction between salicylaldehyde and 2-phenylglycinemethylesterhydrochloride are relatively few (Iorungwa *et al.*, 2020b).

Herein, we have reported the synthesis, characterization, and thermal behavior of Cu^{2+} and Fe^{2+} complexes derived from N-N¹-diphenylhydroxylamineethylacetate as a continuation of our research findings in the area of complexes with azomethine ligands.

MATERIALS AND METHODS

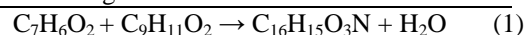
All the chemicals and solvents used for the synthesis were of analytical grades and purchased from BDH and Merck Chemical Co. They were used without further purification. The infrared spectra of the ligand and metal complexes were prepared using DGH-9101-ISA PEC model oven and analyzed using KBr disc in the range 4000-450 cm^{-1} on a Shimadzu infrared spectrophotometer. X-ray power diffraction characterization of the synthesized Schiff base and its metal complexes were carried out in Umaru Musa Yar'adua University Katsina State Nigeria in the central research laboratory with X-ray Diffractometer Thermo Scientific Model ARL X TRA X-ray 197492086.

Preparation of N, N¹-diphenylhydroxylamineethylacetate
The synthesis of N, N¹-diphenylhydroxylmethyleneacetate was carried out

at 50°C for 3 days and preserved in the refrigerator. The Chemistry of reaction leading to the synthesis of the ligand is as presented in Equation (1)

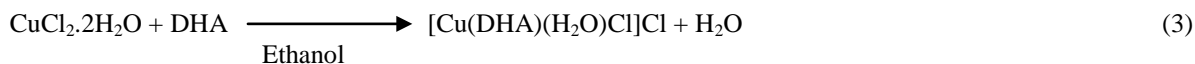
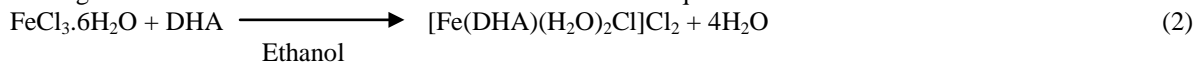
	$\lambda_{\max}(\text{nm})$	$\lambda \text{ max}(\text{cm}^{-1})$	Assignment	Geometry
DHA	225	44.444	π - π^*	Tetrahedral
	265	37.736		
DHA-Cu	230	43.478	Charge transfer	
	252	39.682		
DHA-Fe	272	36.764	Charge transfer	
	271	36.900		

in accordance with the method described elsewhere (Kavitha and Anantha, 2014; Iorungwa *et al.*, 2020). Exactly 3.3 g (0.01 mmol) of 2-phenylglycinemethylester hydrochloride accurately weighed into a crucible was prepared as described previously. Salicylaldehyde (2.5 g) and 10 mL of absolute ethanol were added. The mixture was stirred for 30 min in a beaker and 2.78 g of triethylamine was added and stirred continuously for 5 min and placed in the thermostated microwave oven, and powered source of 220 V for 30 min. Stirring was continued and the temperature of the oven was maintained at 100°C and monitored using a thermocouple device. A bright yellow solid product formed was kept overnight in a refrigerator to cool, it was thereafter filtered, washed with distilled water, recrystallized, washed with again with distilled water and dried in an oven



Synthesis of complexes

Synthesis of the complexes was carried by adopting the methods described by Aswathy *et al.*, (2020) with little modifications. Exactly 5.0 g (0.01 mol) of Schiff base ligand was weighed and 20 mL ethanol added drop wise into 1.0 mmol of metal salts of Cu (II) chloride and Fe (II) chloride respectively in a 250 mL beaker, each mixture was stirred thoroughly and placed into a microwave oven for 30 min. The metal complexes formed were kept in a refrigerator overnight to cool, it was thereafter filtered and washed with distilled water and recrystallized. The products formed was air-dried and preserved in a refrigerator at 50°C. The synthesis of the complexes was done according to Equations 2 and 3.



RESULTS AND DISCUSSION

Tables 1 and 2 present the physicochemical properties and the electronic spectra of the ligand and complexes respectively while Table 3 presents the unit cell parameters of the ligand and complexes. The thermoanalytical data of the complexes has been presented in Table

4 while Tables 5 and 6 show the X – ray diffraction data of the respective complexes. The synthesized compounds were crystalline, coloured and soluble in acetone, DMF, methanol and ethanol as seen in Table 1. The spectroscopic data for the synthesized metal complexes were in agreement with the proposed geometry.

Table 1: Some physicochemical characteristics of ligands and complexes

Ligand/Complex	Molecular Formula	Molecular Weight	Colour	Yield(%)
DHA	$\text{C}_{16}\text{H}_{15}\text{O}_3\text{N}$	269	Yellow	81.4
DHA-Cu	$[\text{Cu}(\text{C}_{16}\text{H}_{15}\text{O}_3\text{N})_2] \cdot 2\text{H}_2\text{O}$	439.48	Deep blue	10.2
DHA-Fe	$[\text{Fe}(\text{C}_{16}\text{H}_{15}\text{O}_3\text{N})_2] \cdot \text{H}_2\text{O}$	467.81	Light brown	6.2

Table 2: Electronic spectral data for ligands/complexes

	$\lambda_{\max}(\text{nm})$	$\lambda \text{ max}(\text{cm}^{-1})$	Assignment	Geometry
DHA	225	44.444	π - π^*	Tetrahedral
	265	37.736		
DHA-Cu	230	43.478	Charge transfer	
	252	39.682		
DHA-Fe	272	36.764	Charge transfer	
	271	36.900		

Table 3: Unit Cell Parameters for Synthesized Ligands and Complexes

S/No	Ligand/Complexes	Unit Cell Parameters			Volume	Crystallite Size	Crystal Structure
		a(Å)	b(Å)	c(Å)			
1	DHA	3.5	5.0	4.6	80.50		Orthorhombic
2	DHA-Fe	4.7	4.0	4.6	86.46	94.74	Orthorhombic
3	DHA-Cu	5.4	6.0	6.1	197.64	35.28	Orthorhombic

Table 4: Thermo Analytical Results of DHA Complexes

Name of complex	TGA (°C)	DTA (°C)	Stage	Mass Cal.	loss	Found	Assignment
DHA-Fe [Fe(C ₁₆ H ₁₅ O ₃ N) ₂].H ₂ O	30-220	50	I	2.7	3.03		Dehydration of 1 mole of water
	221-410	360	II	39.3	35.5		C ₁₄ H ₁₃ O ₂ N
	411-500	480	III	46.0	44.3		C ₁₈ H ₁₇ O ₃ N
	>500	740	IV	11.9	12.1		FeO Residue
DHA-Cu [Cu(C ₁₆ H ₁₅ O ₃ N) ₂].2H ₂ O	30-300	80	I	5.0	5.98		Dehydration of 2 moles of water
	301-398	320	II	37.1	38.21		C ₁₃ H ₁₂ O ₃ N
	400-480	450	III	38.21	44.52		C ₁₇ H ₁₈ O ₂ N
	>480	690	IV	18.1	13.28		CuO Residue

Table 5: X-Ray Diffraction Studies of [Cu(C₁₆H₁₅O₃N)₂].2H₂O Complex

S/No	d-Spacing (Å)		2θ values		Δ2θ	hkl
	Observed	Calculated	Observed	Calculated		
1	5.95315	5.94594	7.4408	7.4291	0.0117	100
2	5.36917	5.36286	8.2554	8.2446	0.0108	100
3	4.21964	4.21455	10.5272	10.5095	0.0177	010
4	3.98870	3.98385	11.1443	11.1279	0.0164	011
5	3.34043	3.33636	13.3436	13.3265	0.0171	111
6	3.20087	3.19701	13.9369	13.9160	0.0209	111
7	3.04579	3.04215	14.6619	14.6432	0.0187	200
8	2.68698	2.68376	16.6734	16.6432	0.0249	220
9	2.58532	2.58224	17.3494	17.3255	0.0239	220
10	2.50754	2.50455	17.9055	17.8785	0.0270	211
11	2.03914	2.03671	22.2141	22.1851	0.0290	300
12	1.99458	1.99218	22.7379	22.7058	0.0321	030
13	1.90254	1.90024	23.9046	23.8723	0.0323	310
14	1.79538	1.79319	25.5295	25.3914	0.1381	311
15	1.60038	1.59976	28.7719	28.7572	0.0147	300

Table 6: X-Ray Diffraction Studies of [Fe(C₁₆H₁₅O₃N)₂].3H₂O Complex

S/No	d-Spacing (Å)		2θ values		Δ2θ	hkl
	Observed	Calculated	Observed	Calculated		
1	4.71624	4.71236	9.40805	9.39268	0.01537	100
2	4.06843	4.06546	10.92315	10.90610	0.01705	010
3	3.53718	3.53373	12.5888	12.5681	0.0207	110
4	3.06603	3.06284	14.5630	14.5426	0.0204	111
5	2.98030	2.97757	14.99165	14.96921	0.02244	111
6	2.81665	2.81329	15.8850	15.8607	0.0243	111
7	2.46702	2.75886	16.20985	18.18583	-1.97601	111
8	2.31503	2.31231	19.45185	19.45185	0.0000	200
9	2.03790	2.03595	22.2284	22.1974	0.0310	210
10	1.99707	1.99481	22.70795	22.67478	0.03317	211

Infrared Spectral Studies

The electronic spectral studies of the ligands DHA, and its metal complexes were recorded in methanol as presented in Table 2. In the DHA ligand, a sharp band appeared at 225 nm corresponding to π - π^* transition, with another band at 265 nm which undergoes a blue shift to the shorter wavelength indicating complexation. The DHA-Fe complex showed a sharp band at lower energy and shifted hypsochromically to an absorption band of 271 nm.

The DHA-Cu complex showed absorption bands at 230 nm. Another broad band appeared at 252 nm which can be attributed to a π - π^* transition. Another absorption band appeared at 272 nm.

The UV-visible absorption spectral studies of the ligands and their metal complexes showed similarities as an indication of the similarities in their structures and geometry.

In the complexes, there were notable changes in both frequencies and intensities in the characteristic bands of the complexes compared to free ligands. The blue shifts and hypsochromic shifts observed in the absorption bands during complex formation indicate coordination of the ligands to the metal ion. These observations are in complete agreement with those of Suman *et al.*, (2015) and Iorungwa *et al.*, (2020a).

FTIR Characterization

The infrared spectral for the ligands and synthesized complexes were recorded using KBr pellets in the range of 4000-450 cm^{-1} and provided

valuable information regarding the nature of the functional groups attached to the metal ion.

The comparison of the FTIR spectral of the synthesized ligand and metal complexes reveals the binding mode of the ligands to the metal ion which is confirmed by the shift in the positions of the absorption peaks as observed in Figure 1. The FTIR results for the complexes are also presented in Figures 2 and 3. The presence of absorption band above 3300 cm^{-1} for the DHA-complexes indicates the presence of water molecules in those complexes as coordinated ligands. A band at 1494 cm^{-1} is assigned to the spectrum ν C=O stretching frequency in the spectrum of the free Schiff base ligand which is shifted to a lower frequency of this band to a lower frequency indicates involvement of oxygen atoms from the COOH which bond to the metal ions. The peaks between 1565-1576 cm^{-1} has been assigned to the ν C=N mode (Hassan, 2015). The shifting of this group to a lower frequency compared to the Schiff base ligand (1576 cm^{-1}) suggest a metal ion coordination through the nitrogen atom of the azomethine group. This band C=N (azomethine) shifted to a lower frequency due to a shift of lone pair density towards the metal centre as collaborated by Abdelsalam, *et al.*, (2019).

The bands observed in the complexes around 1394 cm^{-1} - 1494 cm^{-1} which are in the finger print region (Raza *et al.*, 2013). Bands appearing at 890 cm^{-1} - 904 cm^{-1} and 685 cm^{-1} - 700 cm^{-1} have been assigned to the M-N and M-O respectively (Sani and Siraj, 2020).

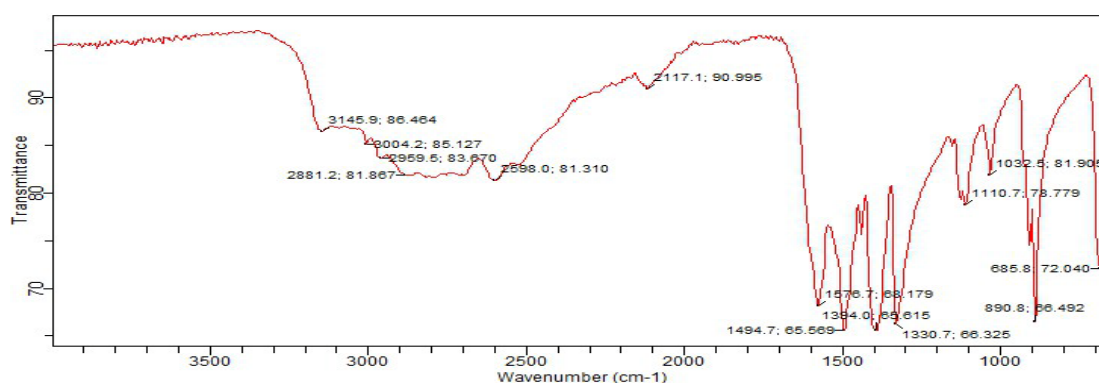


Figure 1: FTIR Spectrum of DHA Ligand

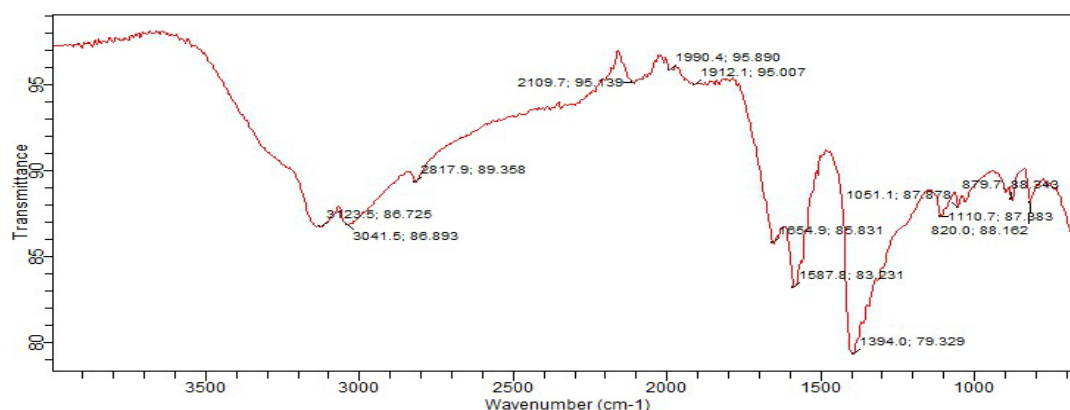


Figure 2: FTIR Spectrum of DHA-Cu Complex

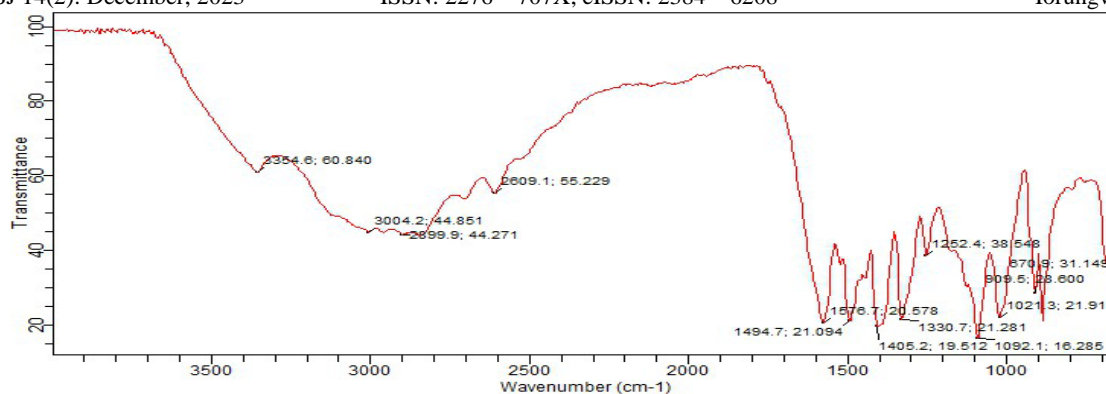


Figure 3: FTIR Spectrum of DHA-Fe Complex

X-ray diffraction (XRD) Characterization

The analysis was carried out to determine the type of crystal system lattice parameters and the cell volume as presented in Table 5-6 and their spectral patterns are in Figures 4, 5 and 6 for the ligand and the complexes.

The XRD pattern indicated a crystalline nature for the ligands and its metal complexes. Indexing of the diffraction pattern was performed by using the trial and error method. The Miller indices (*hkl*) along with observed and calculated 2θ angles, the observed and calculated *d* values are presented in Table 5-6. From the indexed data, (Table 5-6), it was found that the ligands and its metal complexes have orthorhombic structure. The crystal structures of similar type of samples were

reported as orthorhombic (Adelsalam *et al.*, 2019). Additionally, using the diffraction data, the mean crystallite sizes of the ligands and complexes *D* was determined according to the Scherrer Equation presented as Equation 4;

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (4)$$

Where λ is the x-ray wavelength (1.5406 Å) θ is the Bragg diffraction angle and β is the full width at half maximum of diffraction peak (Chandraleke and Chandramohan, 2014; Rabia *et al.*, 2016). The average crystallites sizes of all the samples were found to be (48.28 nm) as presented in Table 5-6.

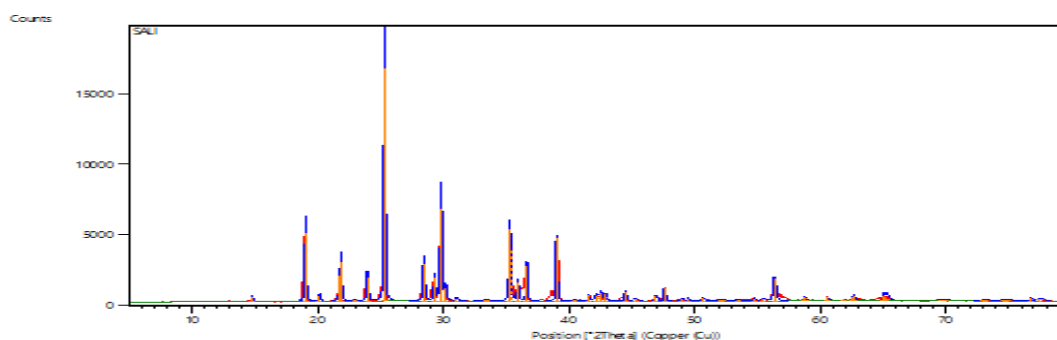


Figure 4: XRD Measurement of DHA Ligand

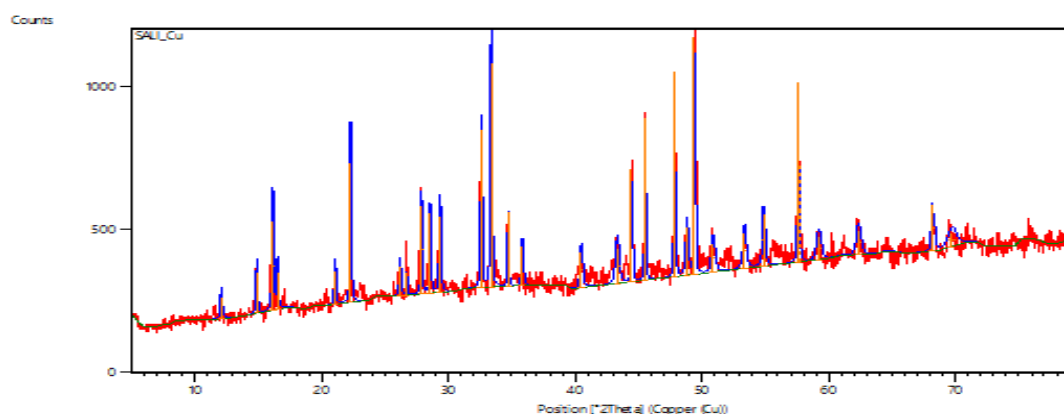


Figure 5: XRD Measurement of DHA-Cu Complex

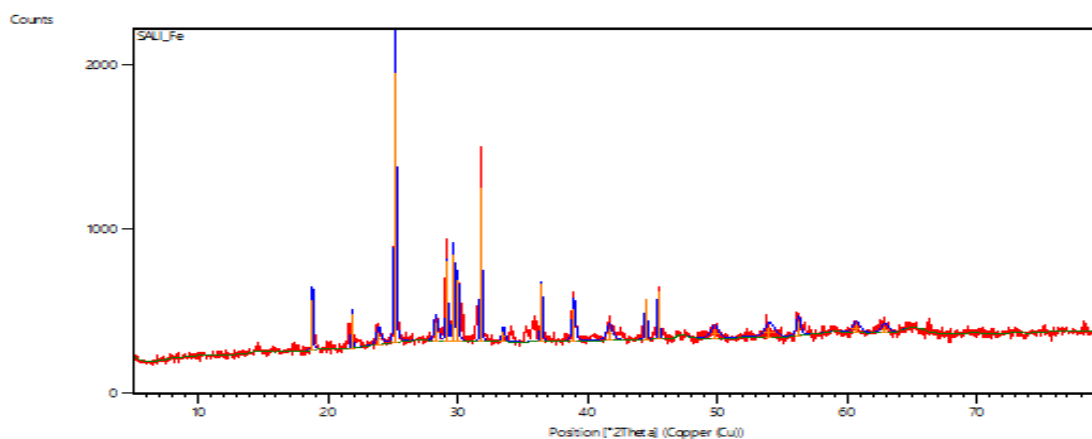


Figure 6: XRD Measurement of DHA-Fe Complex

Uv/vis spectral characterization

In the DHA ligand, a sharp band appeared at 225 nm corresponding to π - π^* transition, with another band at 265 nm which undergoes a blue shift to the shorter wavelength indicating complexation. The DHA-Fe complex showed a sharp band at lower energy and shifted hypsochromically to an absorption band of 271 nm. The DHA-Cu complex showed absorption bands at 230 nm. Another broad band appeared at 252 nm which can be attributed to a π - π^* transition. Another absorption band appeared at 272 nm. The UV-visible absorption spectral studies of the ligands and their metal complexes showed similarities as an indication of the similarities in their structures and geometry.

In the complexes, there were notable changes in both frequencies and intensities in the characteristic bands of the complexes compared to free ligands. The blue shifts and hypsochromic shifts observed in the absorption bands during complex formation indicate coordination of the ligands to the metal ion. These observations are in complete agreement with those of (Hossain *et al.*, 2017).

Thermo gravimetric Analysis

The thermal gravimetric analysis (TGA) and Differential thermal analysis (TDA) experiments were carried out on the complex to determine the thermal stability of the complexes. The thermal behaviours of all the synthesized metal complexes were studied in the temperature range of 30-1000°C. Thermal analysis plays an important role in studying the stability, melting point, structure and decomposition properties of the metal complexes (Pravin *et al.*, 2019). The thermo gravimetric analysis of the metal complexes has been studied to establish different decomposition process and confirm the proposed stoichiometry (Radha *et al.*, 2020).

It also provides information about thermal stability of the complexes and decides whether the

water molecules are inside or outside the inner coordination sphere of the central metal ion. The results of the thermal behavior of the synthesized metal complexes are presented in Table 4. The result indicated a reasonable correlation between the calculated and found weight loss values.

The simultaneous TGA/DTA analysis of all metal complexes were studied where the heating rates were suitably controlled at 10 min under nitrogen atmosphere and weight loss was measured from ambient temperatures to 1000 °C. The decomposition temperature range, percent mass loss and residue leaving behind are presented. The thermo gravimetric analysis of DHA-Fe with the formula $[\text{Fe}(\text{C}_{16}\text{H}_{15}\text{O}_3\text{N})_2 \cdot \text{H}_2\text{O}]$ indicated a weight loss of 2.7 % found and 3.03 % calculated associated with dehydration of 1 molecule of water at the temperature of 30-220 °C. The second decomposition stage proceeded at 221-410 °C corresponding to the loss of $\text{C}_{14}\text{H}_{13}\text{O}_2\text{N}$. Percentage weight loss of 39.2 % found and 35.5 % calculated strongly corrected. The third and fourth stages of decomposition corresponding to $\text{C}_{18}\text{H}_{17}\text{O}_3\text{N}$ and FeO residue at the temperature range of 411-500°C and above 500°C, with weight losses of found 46.0 %, calculated 44.3 %, found 11.9 %, calculated 12.1 % respectively.

The thermo gravimetric curve of the complex $[\text{Cu}(\text{C}_{16}\text{H}_{15}\text{O}_3\text{N}_2)_2 \cdot 2\text{H}_2\text{O}]$ shows a thermal dehydration at a temperature range loss of 5.98 % found, 5.0 % corresponding to the total mass loss of 2 molecules of water. Elimination of $\text{C}_{13}\text{H}_{12}\text{O}_3\text{N}$ occurred at the second stage of the decomposition process at the temperature of 301-398°C with the weight loss of 37.1 % found, 38.21 % calculated. The third stage decomposition suffers the loss of $\text{C}_{17}\text{H}_{18}\text{O}_2\text{N}$ moiety equivalent to the percentage weight loss 38.21 % found and 44.52 % calculated. The ZnO residue decomposed at the temperature of 690 °C in the fourth stage of the decomposition process with the percentage weight loss of 18.1 % found, 13.28 % calculated. In all cases the residues are the metal oxides Joshi *et al.* (2011); Suman *et*

al. (2015). The percentage weight loss in all the complexes is in agreement with the calculated values Kavitha *et al.* (2015). The nature of the thermographs and percentage weight loss in all the complexes correspond to $[ML_2].3H_2O$, $[ML_2].2H_2O$, for DHA-Cu and DHA-Fe respectively. These observations are recorded elsewhere (Aswathy *et al.*, 2020). The percentage

loss in all the complexes is in agreement with all the calculated values. Similar findings were reported by Kavitha *et al.* (2015). The absence of weight loss higher temperatures indicated that there is no hydrated water molecule in the crystalline solid (Fei – Ran *et al.*, 2014). The thermogravimetric curves for the complexes are presented in Figures 7 and 8.

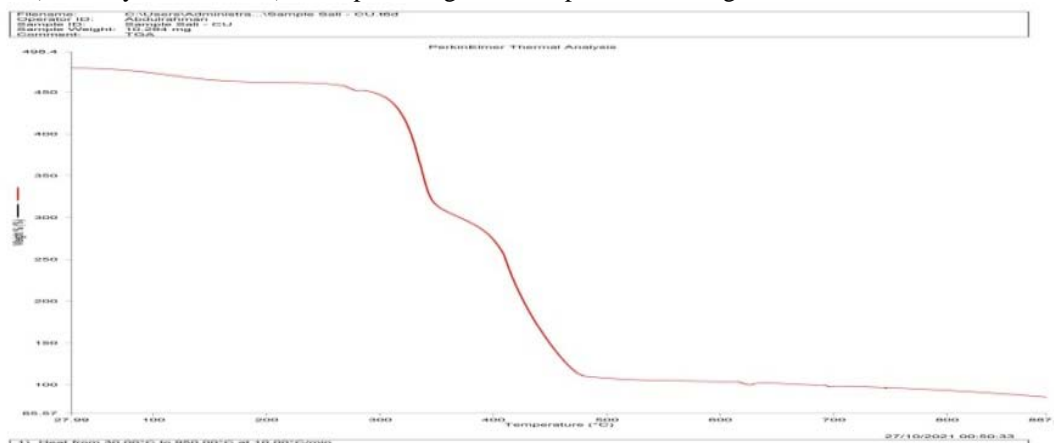


Figure 7: Thermogravimetric curve for DHA-Cu Complex

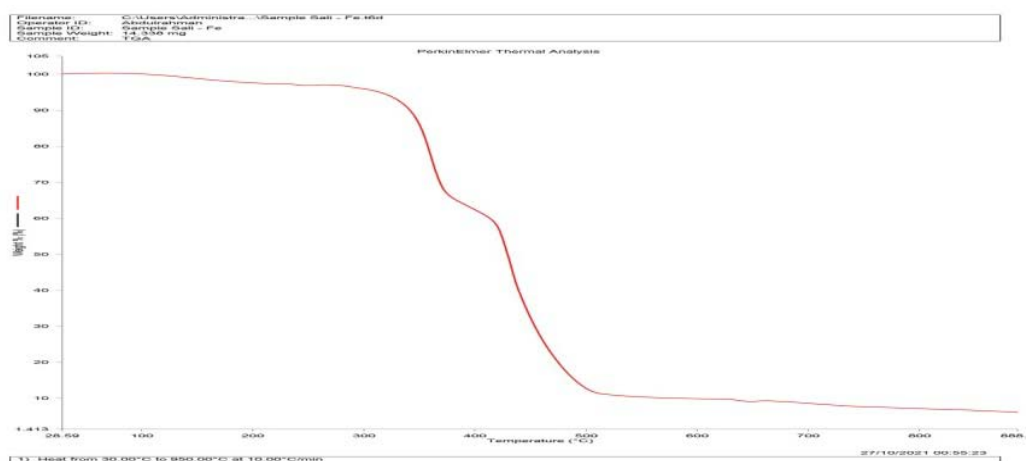


Figure 8: Thermogravimetric curve for DHA-Fe Complex

CONCLUSION

The synthesis of complexes of azomethine linkages have been extensively studied, and it is an area of research with great potentials in new drug design.

The present work describes the synthesis of Cu^{2+} and Fe^{2+} Schiff base complexes derived from the condensation reaction of 2-phenylglycinemethylesterhydrochloride with salicylaldehyde. The physical (molar conductance, magnetic susceptibility measurement and thermal

gravimetric analysis, spectral (uv-vis, FTIR) data for the complexes provides clear evidence that the Schiff base is coordinated to the metal ion through the azomethine linkage. The thermogravimetric analysis affirms the stability of the complexes and the position of the water molecule in the coordination sphere of the complexes. Supporting evidence from the XRD studies agrees with the proposed tetrahedral geometry of the complex (Figure 9).

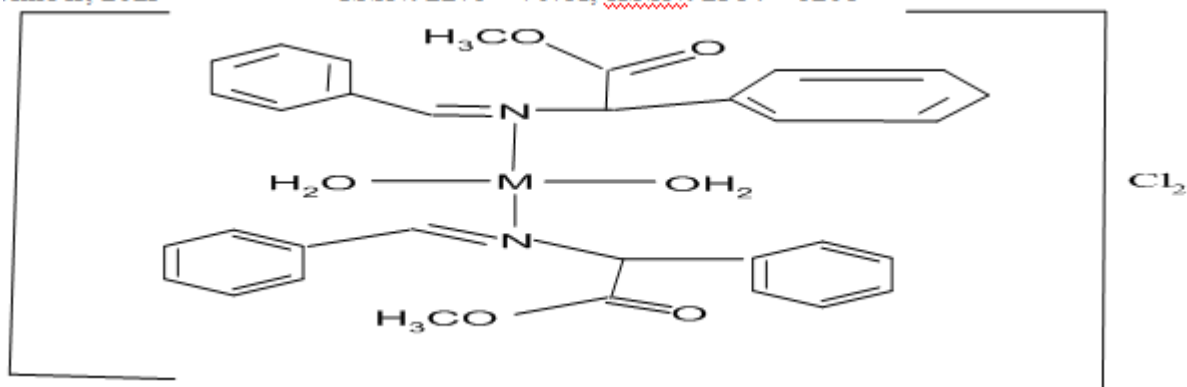


Figure 9: Proposed structure for the tetrahedral geometry of the DHA-Metal complexes

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