SYNTHESES, CHARACTERIZATION AND THERMAL STABILITY STUDY OF NEW Co(II), Cu(II) AND Zn(II) COMPLEXES DEVIATES FROM SCHIFF BASE 4-(DIMETHYLAMINO)BENZALDEHYDE

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Abstract: *Schiff base complexes were known for their various applications in different fields, due to their interesting chemical and physical properties. In this work, Bis(4-dimethylamino)bezylidene) ethanebis(thioamide) (DMABT) as Schiff base ligands was prepared via condensation reaction between 4-(dimethyl amino) benzaldehyde and dithioximade Then, the resulted ligand coordinated with central elements Co(II), Cu(II). and Zn(II). The new complexes are known; [Co2(DMABT)], [Cu2(DMABT)] and [Zn2(DMABT)] were successfully characterized through various techniques such as electrochemical conductivity, magnetic susceptibility, infrared spectroscopy, electronic spectrum measurements, and nuclear magnetic resonance spectroscopy of carbon 13 and proton. In addition, both thermal stability and kinetic parameters were investigated to report data of energy activation reaction, enthalpy, entropy, Gibbs free energy reaction constant, and half-life of thermal decomposition. For the previous proposal, a METTLER Toledo module device was carried out the samples at a 10 °C\min rampage, starting at 25 °C up to 600 °C. According to the reported results, the prepared compounds have tetrahedral shapes with high thermal stability and could be suggested to apply in various future industrial application*

Keywords: *Schiff base, complexes, thermal stability, kinetic, thermodynamic study.*
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1. Introduction

Schiff bases products prepared first time by German chemist Hugo Schiff in 1864 [1]. It can be prepared from direction reaction between aliphatic or aromatic Ketone or aldehydes with aliphatic or aromatic primary amines, as shown in the Fig. 1.

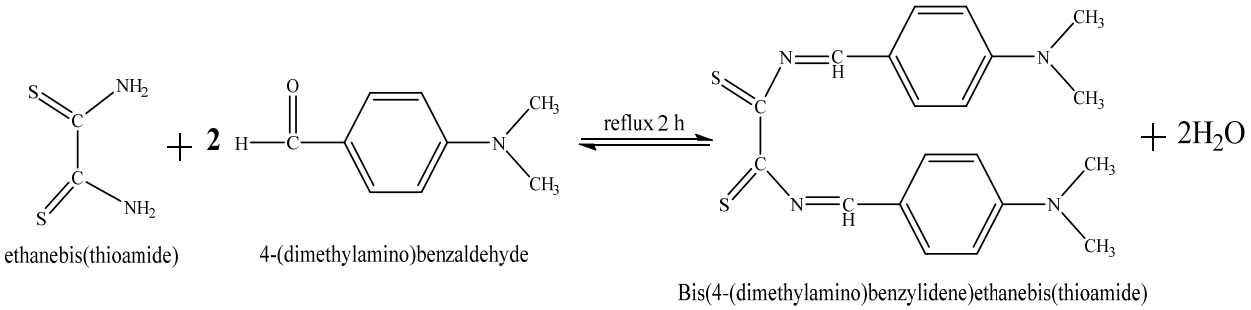

Fig. 1. An example of Schiff base preparation as donor electron (ligand)

For more details, the resulting Carbon – Nitrogen bonds (>C=N) are it known as Schiff base. N atom is considered an electron-donor atom. It's also could be substituted with other electron-donor atoms like oxygen(O) or sulfur(S). Schiff base is widely used in fields of coordination chemistry due to their free election pairs. It is also easily bonded with central

transition elements (accepter electrons) to form stable chemical compounds through mono-, tri-, tetra- or poly-dentate ligands. In addition, Schiff bases form extremely stable complexes with different transition and non-transition elements [2, 3]. Schiff bases in their metal complexes can be applied in different industries and nanotechnology applications such as; the dye industry, microelectronics, optoelectronics, antibacterial, anticancer, biological sensors, agrochemical industries, food industry, photocatalytic energy harvesting, analytical chemistry storage devices, environmental and chemo-sensing applications, biomedical, wastewater treatments and removing heavy metals from aqueous solutions [4-7].

For these reasons, a significant amount of studies have focused on investigating Schiff bases as ligands in the last few decades. The other interesting feature of Schiff base ligands, it can bind to multiple elements with various oxidation states of the central ion metals. Furthermore, Schiff bases when bounded with metal the resulting complex have more thermal stability [8-10].

Schiff bases compounds that have high thermal stability were already carried out as conductive, nonlinear, optical properties and fiber forming applications. Thermal stability and kinetic parameter studies such as entropy, enthalpy, and activation energy calculations can be can be easily investigated through thermogravimetric analysis, with, differential scanning calorimetry.

The thermal stabilities of resulted Schiff base complexes were approved to be more stable than pure Schiff base ligands. For instance, Moftakhar et.al [11] showed that 3-methoxy salicyl aldimine propyl triethoxysilane (MNS1), 5-bromo salicyl aldimine propyl triethoxysilane (MNS2), and 3-hydroxy salicyl aldimine propyl tri-ethoxy silane (MNS3) were more stable when they coordinated with Cu(II) central metals. Their study also investigated both the kinetic and thermodynamic behaviours such as observing the change in entropy, enthalpy, and free energy of Gibbs, the kinetics of decomposition. The study confirmed the stability of Schiff bases complexes [11]. Other research was done by Ejidike group [12]. When they compared the thermal stability of the same Schiff base ligands with different transition elements as control parameters, They concluded thermal stability of Schiff base complexes was following sequential stability Ni(II) > Cu(II) > Co(II) > Fe(II) [12].

Recently, Hossain, et. al. coordinated Mn(II), Fe(II), Co(II) and Cd(II), with, ligand 4((pyridin-2-ylimino)methyl) phenol. The reported results revealed moderate to strong antibacterial activities against some types of bacteria like Bacillus subtilis and Staphylococcus aureus [13]. Another research done by Ahmadi, et. al. [14]. They firstly, prepared N,N'-Bis(4 dimethylaminobenzylidene)benzene-1,3-diamine as schiff base via reflexed 4-dimethylaminobenzaldehyde with 1,3-phenylenediamine. Then, coordinated the ligand with Zn(II), Cd(II) Ions. They reported that prepared complexes have sufficient antibacterial effect on Staphylococcus aureus, Escherichia coli, and Bacillus subtilis.

In this work, a new Schiff bases ligand is synthesized by condensation reaction of 4-(dimethyl amino) benzaldehyde with with dithioximade. Then, the resulted ligand, Bis(4-dimethylamino)bezylidene)ethanebis(thioamide) (**DMABT**) was coordinated with Co(II), Cu(II), and Zn(II)to form new [Co_2(DMABT)], [Cu_2(DMABT)] and [Zn_2(DMABT)] Schiff complexes. The complexes have been successfully characterized by different techniques such as Fourier Transform Infrared Spectroscopy (FT-IR), Proton nuclear magnetic resonance (^{1}H-NMR), and Carbon-13 nuclear magnetic resonance(^{13}C-NMR), uv-vis absorbance and elemental analyses.

The aim of this research was to invesigate the thermogravimetric analysis (TGA) including activation energy, entropy, enthalpy, and Gibbs free energy, a kinetic analysis (half-life, reaction constant), and thermal stability of these preapred complexes. The shown results fistly, confirmed the stabilty of Schiff base complexes at high temperature. All compexes showed endothermic reaction Behavior during decompsion. While kinetic reaction constant (K) was in first order reaction type in range (0.013-0.087) K (min-1) and half-live time ($T_{1/2}$) range 19.03-52.10 min.

2. Experimental part

2.1 Chemiacal materials. All chemical materials; Copper chloride(II) hydrates ($CuCl_2.2H_2O$), Cobalt chloride(II) hydrates ($CoCl_2.6H_2O$), and Zink Chloride($ZnCl_2$), 4-(dimethyl amino) benzaldehyde, and solvents such as; Ethanol, Dimethyl sulfoxide(DMSO), were provided from different companies like fluka, Merch and Sigma-Aldrich without further purification.

2.2 Preparations of the compounds. All compounds prepared as well as in common methods in previous works of literatures [15, 16].

Synthesis of Schiff base ligand (DMABT) N^1,N^2-bis(4-dimethylamino) benzylidene) ethane bis (thioamide): 4-(dimethyl amino) benzaldehyde (2.98 g, 0.02 mol) was dissolved in 20 ml of absolute ethanol and mixed with dithioximade (1.20 g, 0.01 mol) dissolved above in (10 ml) absolute ethanol. The mixture then was heated and stirred under reflux for 2 hours with bar magnetic stirrer. Finally, brown precipitate was formed after cooling at room temperature for 24 hours in high yield (98%) filtered off, washed by distilled water and diethyl ether then dried under Vacuum for several hours**.**

Preparation of complexes [M_2L]: A clear solution of the ligand N1,N2-bis(4-dimethylamino) benzylidene) ethane bis (thioamide) (0.38g, 0.001 mol) in ethanol (20 ml) was added to a solution of $CoCl_2.6H_2O$, $CuCl_2.2H_2O$ or $ZnCl_2$ (0.002 mol) in (10 ml) ethanol, the reaction mixture was refluxed for 2 hours with magnetic bar stirrer. Then the mixture was left for 24 hours, at room temperature to obtain the precipitate which was filtered off and washed with diethyl ether. Then dried under vacuums for 3 hours.

2.4 Characterizations of prepared compounds. A variety of physical and spectroscopic techniques were used to thoroughly characterize all of the prepared compounds;

Melting Point or Decomposition Temperature Measurement of melting or dissociation points of all prepared compounds was carried out on an Electrothermal 9300 Model

Conductivity Measurement: dimethyl sulfoxide (DMSO) 10^{-3} Molar solutions were carried out on a SIBATA Conductivity Meter Model SC 17A, at room temperature.

Magnetic Measurement: The magnetic susceptibility of the complexes prepared at room temperature was evaluated using the Gouy Method. A Magnet Bruker (B.M6) device was used for this purpose. The magnetic correction factor (D) was also calculated using Pascal's constants. Specific to the atoms that make up the prepared complexes.

Electronic Spectra: Electronic Spectra Ultraviolet radiation measurements of the complexes were recorded by Shimadzu UV visible spectrophotometer module UV 1601. 200-1100.

Proton nuclear magnetic resonance (^{1}H-NMR) and Carbon-13 nuclear magnetic resonance (^{13}C-NMR). Hexadeutrodimethyl sulfoxide (DMSO-d6) solvent was used as Schiff base complexes solvent for Proton nuclear magnetic resonance (**^{1}**H-NMR) and Carbon-13 nuclear magnetic resonance (**^{13}**C-NMR) for spectrophotometer investigation. Bruker Avance DPX 400 MHz) studies. spectrophotometer in the 400-4000cm^{-1} range using the KBr matrix.

Elemental analysis: Thermo Electron Corporation's Flash EA 1112 Series was used to measure the elemental analysis of the complex.

Atomic absorption: Bruker alpha FT-IR was used to generate an infrared spectrum. SensAA GCB scientific equipment system (Avanta 2.02 software) was used to measure the atomic absorption.

The electronic spectrum: Shimadzu 1800 spectrometer UV-Vis; was used to measure the electronic spectrum of ligand and complexes in range 200-1100 nm. DMSO 10^{-3} molar was the solvent of prepared complexes.

Fourier-transform infrared spectroscopy(FT-IR): The samples carried out using SHIMADZU FTIR-8400 spectrophotometer in range 400-4000 cm-1 using KBr matrix.

Thermogravimetric analysis: METTLER TOLEDO TGA\DSC and STARe evaluation software virgin (16.01) were used to examine the thermal parameters. The samples was exposed to heat in range of at 25–600 °C with a ramping heat rate of 10 °C/min under air.

3. Results and Discussion

3.1 Characterization of ligand and complexes. The physical data and quantitative analysis of prepared compounds are displayed in Table 1.

Table 1. Physical data and quantitative analysis of the prepared compounds

No.	Compounds	Colour	m.p. °C	Conductivity DMSO, S^{-1}	Elemental analysis Calculated / (Found)			
					C%	H%	N%	M%
1	L = (**DMABT**) $C_{20}H_{22}N_4S_2$	Light brown	135-140	39	62.80 (62.52)	5.80 (5.82)	14.65 (14.61)	---
2	**[Co_2(DMABT)]** $C_{20}H_{22}Cl_4Co_2N_4S_2$	Light black	246-254	51	37.41 (37.36)	3.45 (3.42)	8.72 (8.67)	18.35 (18.33)
3	**[Cu_2(DMABT)]** $C_{20}H_{22}Cl_4Cu_2N_4S_2$	Dark grey	300>	50	36.88 (36.84)	3.40 (3.35)	8.60 (8.58)	19.51 (19.45)
4	**[Zn_2(DMABT)]** $C_{20}H_{22}Cl_4Zn_2N_4S_2$	Brown	285-287	63	36.67 (36.62)	3.39 (3.32)	8.55 (8.49)	19.96 (19.92)

The melting point of Schiff base complexes were in range (246-300) °C which its higher than pure ligand L = (DMABT) due to the stability of complexes. On the other hand, the electrical conductivity range was in range (39-63) S-1. Suggested the coordination ratios of (Metal-Ion: halogen) be (1:2). While the Elemental analysis of carbon(C), Hydrogen(H) and Metal(M) calculated ratios were found close to practical measurements as can be seen in Table 1.

The compounds were prepared according to the suggested general reaction scheme (Fig. 2), and then these compounds were characterized by spectroscopic and other physical methods.

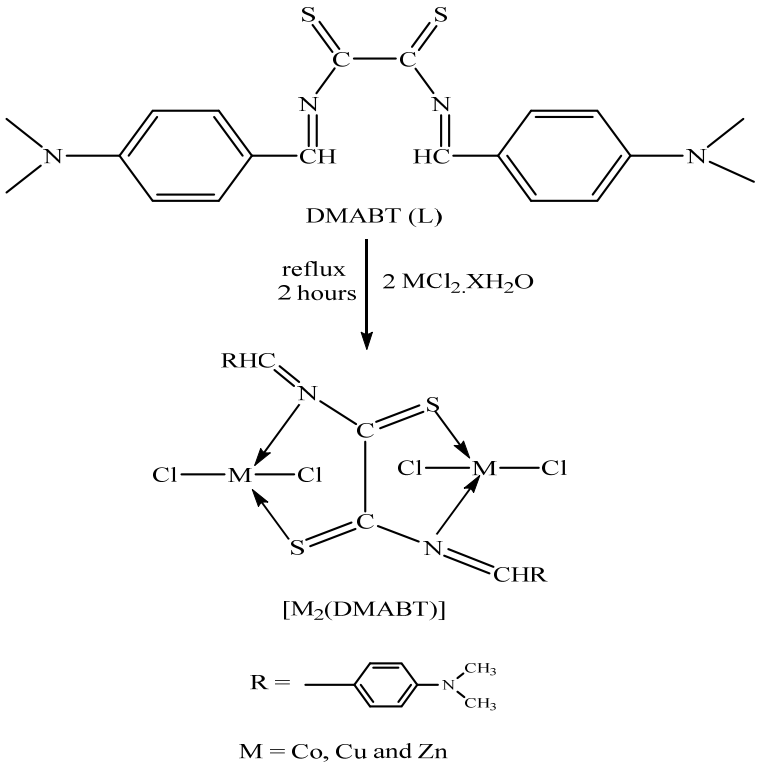

Fig. 2. General reaction scheme to prepare the Co, Cu and Zn complexes

The general reaction diagram illustrates how the ligand binds to the central metal ion in a tetrahedral shape. The shape is confirmed by measurements of ultraviolet, infrared electronic spectra and μeff. Bohr magneton measurement [11, 14, 15] as can be discussed soon.

The coordination complexes' magnetic susceptibility and electrical conductivity measurements, which are displayed in Table 2, both supported these findings.

Table 2. Spectral characteristic and Magnetic data of prepared compounds

No.	Compounds	μeff. (B.M.)	υ(C=N) cm^{-1}	υ(C=S) cm^{-1}	υ(M-N) cm^{-1}	υ(M-S) cm^{-1}	υ(M-Cl) cm^{-1}	d-d transition cm^{-1}
1	L = (DMABT)	---	1649	846	---	---	---	30211
2	[Co_2(DMABT)]	4.5	1535	771	466	424	410	26000
3	[Cu_2(DMABT)]	1.95	1525	815	486	437	415	13260
4	[Zn_2(DMABT)]	Dai	1602	817	522	438	420	-----

The ligand was successfully identified by HNMR spectra of the proton and carbon-13, as well as other measurements, these results confirm the proposed chemical structure of the ligand (**DMABT**) as shown in Fig. 3 and 4. ^{1}H-NMR spectra results are summarized as follows: ^{1}H-NMR (DMSO-d_6, 400 MHz): δ2.990 (12H-4CH_3, s), δ6.773-6.796, δ7.676-7.698 (8H-Ar, m), δ9.622 (2H-CH, s).

For more details, the peaks at 2.99 ppm related to twelve protons of four methyl groups of the ligand (**DMABT**). While the peaks at δ6.773-6.796, δ7.676-7.698 ppm associated to eight protons of aromatic structures. Finally, the peak at δ9.622 related to the two protons of (C-H) groups.

While ^{13}C-NMR results summarized as flowing (DMSO-d_6, 400 MHz): δ39.93,40.11 (A, 4CH_3, d), δ111.52-11180 (B, CH-Ar, d), δ124.95 (C, C-Ar, s), δ132.09 (D, CH-Ar, s), δ154.66 (E, C-Ar, s), δ190.36 (F, CH, s), δ194.89 (G, C-amine, s). The peak at δ39.93 refers to Carbon of four (CH_3) groups. While δ111.52-11180 peaks belonged to one of (CH) group that associated with aromatic structure. δ124.95 peaks sited for second carbon in in (CH) group that associated aromatic structure.

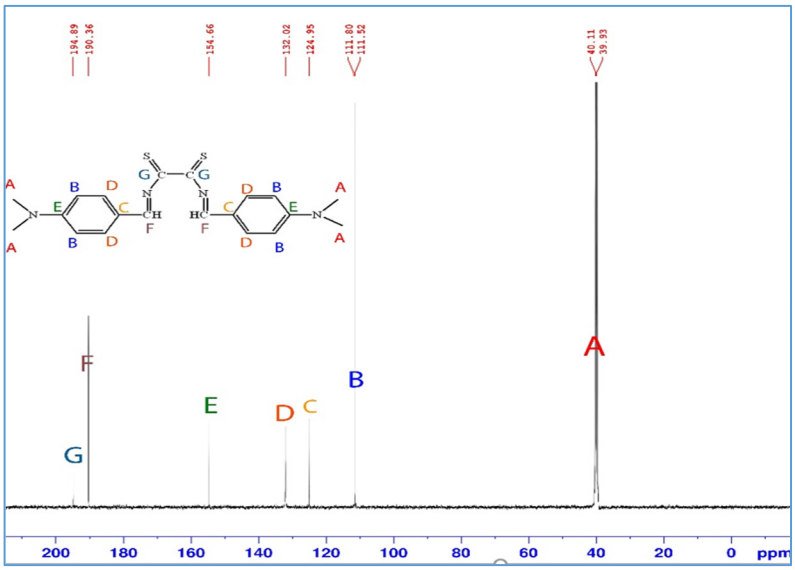

Fig. 3. The ^{1}H-NMR of the ligand (DMABT)

The reported values of μeff. Bohr magneton (B.M.) for [Co_2(DMABT)], [Cu_2(DMABT)] and [Zn_2(DMABT)] were 4.5, 1.95, dai (B.M) respectively. According to these results it was suggested that all complexes were tetrahedral [17]. For more μeff. (B.M.), of [Co_2(DMABT)] showed high spin electron configuration d^7 $(t_2g)^5 \rightarrow (eg)^2$ which is also confirmed by (d-d) electronic spectra with band at 26000 cm^{-1} that belong to allowed electron

transition from $4A_2g$ (F)→$4T_2g$ and $4A_2g$ (F)→$4T_2g$ (F) respectively [18]. All these results supported tetrahedral shape of [Co_2(DMABT)] [19]. While μeff. (B.M.) of [Cu_2(DMABT)] value at 1.95 (B.M) showed electronic spin $d^9 \rightarrow t_2g)^6 \rightarrow (eg)^3$ [20]. Its electronic spectra bands 23866 cm^{-1} showed one allowed electronic transition has one state to state transition $2Eg \rightarrow 2T_2g$ and its band around 13260 cm^{-1} [21]. Finally, the complexes [Zn_2(DMABT)] has d10 diamagnetism and has not (d-d) electronic transition [19, 22].

The ligand (DMABT) also showed another peak at (846) cm^{-1} which is often related to υ(C=S) group. The ligand when associated with central metal ion, this peak was shifted to lower frequency to appear at (771) cm^{-1}, (815) cm^{-1} and (817) cm^{-1} for Co(II), Cu(II) and Zn(II) complexes respectively. These result consistent with what was published in the literature [24]. Other peaks appeared at (466-522) cm^{-1}, (424-438) cm^{-1} and (410-420) cm^{-1} belongs to υ(M-N), υ(M-S) and υ(M-Cl) consequently [25]. It was suggested that Schiff base prepared complexes' structure is tetrahedral, which consistent with previous similar studies [26-28].

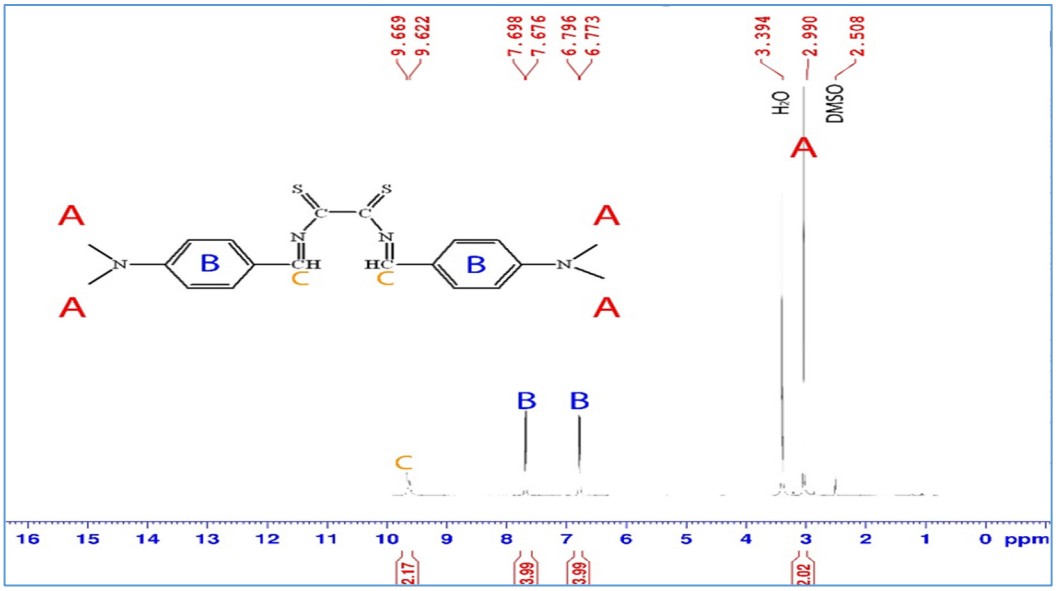

Fig. 4. The ^{13}C-NMR of the ligand (DMABT)

Table 3. The prepared compounds' thermal decomposition percentage

No.	Compounds	1st decomposition	2nd decomposition	3rd decomposition
1	L = (DMABT)	20%	21%	30%
2	[Co_2(DMABT)]	15%	15%	50%
3	[Cu_2(DMABT)]	16%	15%	10%
4	[Zn_2(DMABT)]	10%	15%	25%

It was observed that all compounds undergo three primary stages of thermal decomposition, as indicated in Table 3. 20% of the ligand (DMABT) decomposed earlier in the first stage, while 15% and 30% of ligand decomposed in the second and third stages respectively. These ratios vary amongst complexes; it was reported that [Cu_2(DMABT)] complex is more stable in the first stage, whereas the [Co_2(DMABT)] and [Zn_2(DMABT)] complexes were more stable in the last stage. The reason for these difference related to the type of central ion-metal that causes variables in thermal stability and causes to differ in dissociation rates. The interesting observation note that ligand (DMABT) has higher decomposition rates among the prepared Schiff base complexes.

3.1 Thermogravimetric data study. As can be seen in Fig. 5, there were, generally, three-phase transmissions for each line. The first phase curve belongs to the dehydration process of chemical compounds for both ligands and complexes. The second phase curve is related to the decomposition of organic chemical materials in ligands. The third phase curve showed the removal crystallization of water in ligands [29, 30]. Due to the straight line for each phase, it was assumed that the three-phase transmission (dehydration, decomposition and condensation) was followed by 1st order reaction. Thus, all thermodynamic parameters such as activation energy (a.e), entropy change (ΔS), enthalpy change (ΔH) and Gibbs energy change (ΔG) were calculated via the TGA data sheet (as given in Equation 1) [31, 32].

$$\frac{dx}{dt} = k(1-x) \qquad (1)$$

$$X = \frac{Wi-Wt}{wi-wf} \qquad (2)$$

Where **Wi** is the initial weight, **Wt** is the sample weight at a particular time. While **wf** is the final weight.

Equation (1) can be rewritten as follows

$$\ln(1-x) = -kt \qquad (3)$$

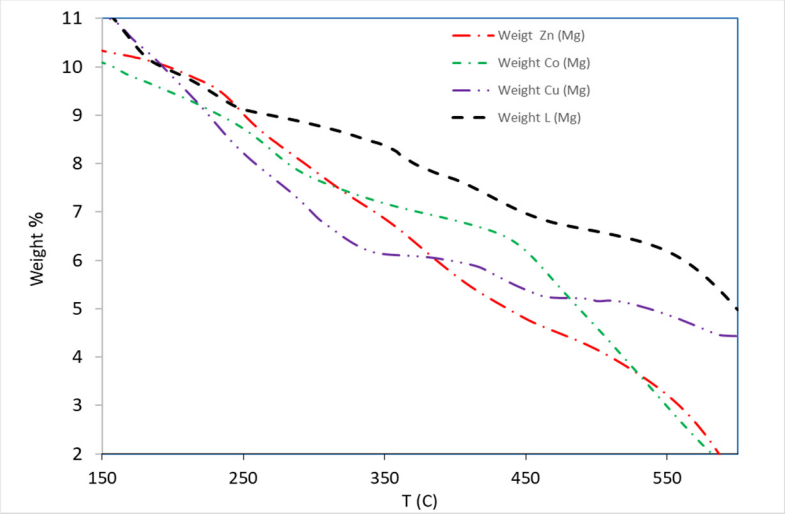

Fig. 5. TGA-DSC curves of complexes and ligand (DMABT)

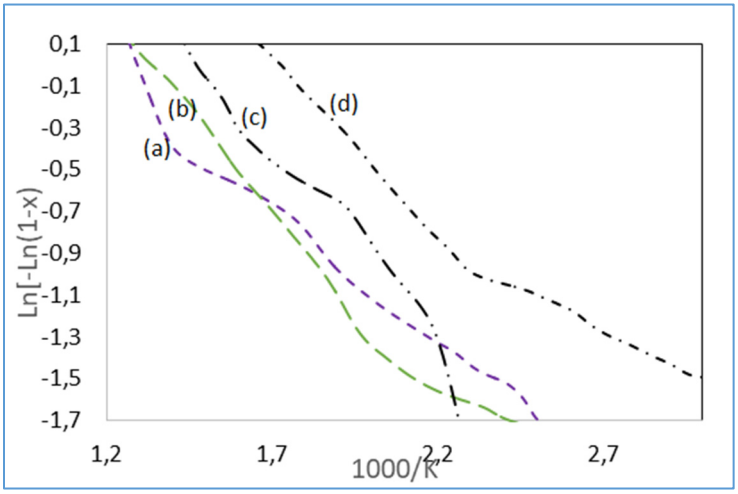

Fig. 6. Plot of ln[-ln(1- x)] vs 1000/T of a) Co, b) Zn, c) L and d) Cu including the three-phase dehydration phase, Decomposition phase and (c) removal water crystallization phase.

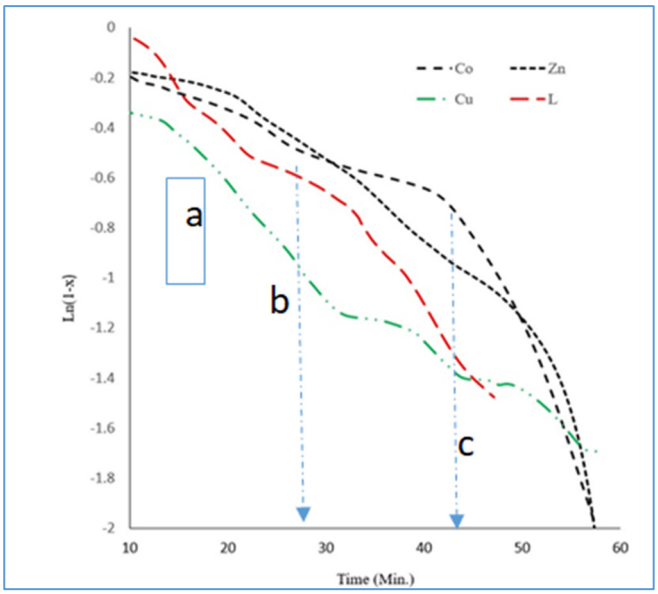

Fig. 7. Plot of ln(1 - x) vs T (min): (a), (b), and (c) are dehydration, decomposition and (c) removal water crystallization phases of Co , Zn , Cu and L complex.

According to equation three, the slope of each phase can determine the rate constant (K) of the chemical reaction. While the half-time ($T_{1/2}$) can be calculated from Equation (4):

$$t_{1/2} = \frac{0.693}{K} \qquad (4)$$

Other kinetic parametrizes can be calculated from the modified coats reform model as described in eq. (5):

$$Ln[-Ln(1\text{-}x) = Ln\frac{ARt^2}{\beta Ea} - \frac{Ea}{RT} \qquad (5)$$

Where is (β) is the heating rate (10 °C /min), (R) is the Gas general constant (8.3143) $J{\cdot}mol^{-1}{\cdot}K^{-1}$. T is the temperature in Kelvin (K). E_a is activation energy. E_a can be determined by plotting $Ln[-Ln(1\text{-}x)$ vs 1000/T (Fig. 6). other thermodynamic calculation was determined as described in previous papers [31, 32].

According to the reported data in Table 4, the dehydration, decomposing and removal crystallization phases started earlier in the Co, Cu, and Zn complex than ligand (except decomposition of the Cu complex and Removal crystallization water in the Co complex (Fig. 7). This may be related to the strength interaction between metal ions and their ligands. While speed reaction constant (K) values in ligands were higher in Ni and Cu complexes. While the lowest (K) value was observed in Co Complex [31, 33]. The lowest $T_{1/2}$ was observed in the Removal crystallization water of the Ni complex and the highest $T_{1/2}$ value was observed in the decomposition phase of the Co complex. In general, it was noted that $T_{1/2}$ values in complex obtained higher than ligand [34].

Table 4. Parameters of each phase's kinetics and thermodynamics during the thermogravimetric analysis of prepared compounds

Ligand L = (DMABT)							
Phase	Temp., K	K (min^{-1})	$T_{1/2}$ (min)	Ea, $J{\cdot}mol^{-1}{\cdot}10^5$	ΔH, $J{\cdot}mol^{-1}{\cdot}10^4$	ΔS, $J{\cdot}mol^{-1}{\cdot}K^{-1}{\cdot}10^2$	ΔG, $J{\cdot}mol^{-1}{\cdot}10^4$
Dehydration	250	0.036	19.03	0.30	2.850	-2.73	9.65

Decomposition	360	0.024	28.51	0.12	0.092	-2.96	11.59
Removal crystallization water [Co_2(DMABT)]	590	0.054	12.76	0.07	0.025	-3.14	19.09
Phase							
Dehydration	160	0.014	48.46	1.59	1.37	-2.47	1.58
Decomposition	340	0.013	52.10	1.14	9.14	-2.74	1.11
Removal crystallization water [Zn_2(DMABT)]	600	0.087	7.96	1.81	6.68	-2.71	1.76
Phase							
Dehydration	140	0.024	28.87	0.10	0.93	-2.80	4.85
Decomposition	340	0.027	25.66	0.12	0.94	-2.88	1.07
Removal crystallization water [Cu_2(DMABT)]	580	0.057	12.15	0.11	0.63	-3.01	1.80
Phase							
Dehydration	220	0.046	15.06	0.064	0.45	-2.98	7.02
Decomposition	460	0.024	28.87	0.162	1.23	-2.96	1.48
Removal crystallization water	590	0.025	27.72	0.116	0.67	-2.99	1.83

The thermodynamic trends among Co, Cu and Zn complexes depends on metal ion size, their charge, and, the nature of their coordination atoms with Nitrogen or oxygen atoms in ligand. But, in general, it was observed that activation energies of complex phases were higher than relative ligands phase due to the stability of complexes if compared with ligands (except dehydration phase in Ni and Cu complexes (they were higher may be due to their smaller ions or connection nature with their ligand). This means the required active energy for phases progresses in complex more than pure ligands [31, 33].

According to positive delta enthalpy values (ΔH), all chemical phases are endothermic reactions. Which means an amount of heat is required to pass the phase reaction forward. For more details, the heat temperature was needed for decomposing and removing water phases in complex more than that in relative phases in ligands. The dehydration phases in complexes are reported to need less an amount of heat if compared with ligands. on the other side, Entropy change (ΔS) values in both ligand and complex were close to each other. Their range was (- 2.47)-(-3.14) $J{\cdot}mol^{-1}{\cdot}K^{-1}x10^2$. Finally, the change in Free Gibbs energy (ΔG) was reported to reduce from ligands to Co, Cu, Zn respectively may be related to the stability of complexes (Table 4).

4. Conclusions

The new Schiff base complexes are successfully prepared and characterized and suggested to have a tetrahedral shape based on earlier findings. The thermodynamic parameters investigation confirmed the stability of these complexes. All chemical phases are endothermic reactions. The activation energy (Ea), enthalpy energy (ΔH), entropy (ΔS) and Gibbs free energy (ΔG) were increased in these heating reactions. The kinetic study of complexes heating was approved that complexes reaction followed a first-order rate equation with kinetic reaction constant (K) range (0.013-0.087) K (min^{-1}) and half-live time ($T_{1/2}$) range (19.03-52.10 min).

Author Contributions: Conceptualization Enas H. mohamed. and Mohammed Alsultan; methodology, resources, project administration, and data curation, Saba M. Alasalli; software, and validation, Assim A. Sabah; formal analysis, writing original draft preparation, supervision and investigation. All authors have read and agreed to the published version of the research manuscript.

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