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SYNTHESIS, SPECTRAL, THERMAL DECOMPOSITION, KINETIC AND ANTIMICROBIAL STUDIES OF AZO-SCHIFF BASES

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ABSTRACT

In this work we focused on the synthesis of new Azo-Schiff bases which were prepared by condensation of 3-formyl-4-hydroxy phenylazo benzene with both 1,4-Bis (3-aminopropyl) piprazine, thiocarbohydazide respectively. The Schiff bases were analyzed by different spectral techniques (LC-MS, GC-MS, 1H-NMR, IR). The new Azo-Schiff Bases were studied for antibacterial activities against gram positive (Bacillus subtilis and Staphylococcus aureus) and gram negative (Salmonella typhi and Escherichia coli) agents. The azo compound ligand exhibited a variable activity of inhibition on the growth of the bacteria. The compounds were subjected to simultaneous thermogravimetric analysis to study their decomposition mechanism and thermal stability.

Keywords: *Azo Schiff Base, 3-formyl-4-hydroxy Phenylazo Benzene, Spectral Techniques, Antibacterial Activity, Thermogravimetric Analysis.*

1. INTRODUCTION

Schiff bases are compounds which are prepared by the condensation of aldehyde or ketone with primary amines [1-3]. They are used in the synthesis of some industrial, bioactive and inorganic compounds [4-5]. The compounds generally show good fungicidal [6], Anticancer [7-9], antibacterial [10-12], antifungal [13,14], anti-inflammatory [15-17] and herbicidal properties [18]. Azo compounds are widely used as dyes and pigments. Another application is analytical chemistry. On the other hand azo compounds show biological activities if it contains antibacterial [18] Schiff bases and thereby azo compounds are important structures in the medicinal and pharmaceutical fields [19]. Azo compounds, with two phenyl rings separated by an azo (-N=N-) bond, are versatile molecules and have received much attention in research areas both fundamental and applied [20]. The strong electronic absorption maximum can be tailored by ring substitution to fall anywhere from the ultraviolet to red-visible regions, allowing chemical fine-tuning of color [20]. This, combined with the fact that these azo groups are relatively robust and chemically stable, has prompted extensive study of azobenzene-based structures as dyes and colorants. Furthermore, the light-induced interconversion allows systems incorporating azo group to be used as photo switches, effecting rapid and reversible control over a variety of chemical, mechanical, electronic and optical properties [21].

Recently heterocyclic azo compounds have been used in the Mitsunobu reaction [22]. Usually, azo compounds were synthesized by diazotization of the amine in mineral acid at

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about 0°C [22-26]. Schiff bases are used as substrates in the preparation of a large amount of bioactive and industrial compounds [27-31]. Azo compounds have a great biological activity as well as industrial importance [32,33]. The interaction of metal complexes with biological systems, the field of biocoordination chemistry, is receiving an increasing interest in recent days [34].

2. EXPERIMENTAL

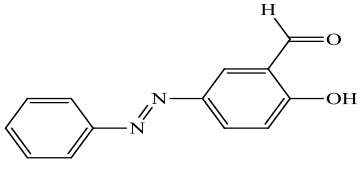
2.1. MATERIALS AND METHODS

Ethanol, dimethyl formamide, salicylaldehyde, aniline, 1,4-[Bis(3-aminopropyl)] piprazine, thiocarbohydazide, Nutrient gar, Filter paper discs (Whatman No.1 filter paper, 5mm diameter) were obtained from Sigma Aldrich Company. IR spectra were recorded on Jasco 300 FT-IR Spectrometer using compressed KBr discs. Mass spectra of the compounds were measured on a micro mass Quattro LC-MS/MS, GC-MS/MS Spectrometer. ^{1}H-NMR spectra were recorded at ambient Bruker DT-400 Spectrometer using $CDCl_3$ with DMSO- DMF as the internal standard, M.P Apparatus Digital (32-300^{0}C). The thermogravimetric analysis (TGA) was carried out in a dynamic nitrogen atmosphere (20 mL.min^{-1}), with a heating rate of 10°C min^{-1} using Shimadzu TGA-50H thermal analyzer.

2.2. SYNTHESIS OF SCHIFF BASES

2.2.1 Synthesis of 3-formyl-4-hydroxy phenylazo benzene (scheme 1)

Azoaldehyde was prepared from aniline and 2-hydroxy benzaldehyde in aqueous medium at 0-5^{0}C according to a reported method and was purified by recrystallization from warm 95% ethanol [35].



Scheme 1 3-formyl-4-hydroxy phenylazo benzene

2.2.2 Synthesis of Bis-N-[(5-phenylazo-2-hydroxy-1-benzylidine)- 1,4-Bis(3-aminopropyl)] piprazine (I) Scheme (2)

Azo-coupled precursors were prepared as described previously [35] was added (0.01 mol) to 3-formyl-4-hydroxy phenylazo benzene in 30 ml from absolute ethanol to 1,4-Bis(3-aminopropyl)]piprazine, (0.005mol) in 60 ml ethanol, The reaction mixture was refluxed for 2 hour with continued stirring. A solid mass was formed in hot. The reaction mixture was allowed to cool to room temperature, it was suction filtered and recrystallized from suitable solvent, then dried in vacuum.

Orange; Yield: (75 %), m. p = 227 - 228^{0}C, Empirical formula: ($C_{36}H_{40}N_8O_2$), M. Wt: (616).

Mass spectrum (m/z): 616 (Fig.1.).

UV-Vis: λ_{max}= 355 nm (Fig.2.).

IR (KBr disk): 3417.86-3475.73 cm^{-1}(O-H), 3039.81-3037.68 cm^{-1} (C-H), aromatic), 2812.21-2935.66 cm^{-1} (C-H), aliphatic), 1635.64 cm^{-1} (C=N), 1585.49 cm^{-1} (C=C, aromatic), 1473.62-1496.76 cm^{-1} (-N=N-), 1284.59 cm^{-1} (C-O), phenolic) (Fig.3.).

^{1}H-NMR ($CDCl_3$-400MHz) δ= 14.28 (s, 2H, OH), 8.449 (s, 2H, CH=N), 7.054 -8.021 (m, 16 H, Ar), 3.704-3.736(t, 4H-CH_2-N=), 2.317-2.520 (t, 4H,-N-CH_2-), 1.710-1.979 (t, 4H,-CH_2-) (Fig.4.).

Elemental analysis: $C_{31}H_{23}N_7O_2$, Calculated (%): C: 70.11; H: 6.54, N: 18.17; O: 5.19 Found (%) C: 70.12, H: 6.49, N: 18.20, O: 5.19.

2.2.3 Synthesis of Bis-N-(5-phenylazo-2-hydroxy-1- benzylidine)-thiocarbohydazide (11) Scheme (3)

Azo-coupled precursors were prepared as described previously [35] was added (0.01 mol) of 3-formyl-4-hydroxy phenyl azo benzene in 30 ml from absolute ethanol tohiocarbohydazide (0.005 mol) in 60 ml ethanol. The reaction mixture was refluxed

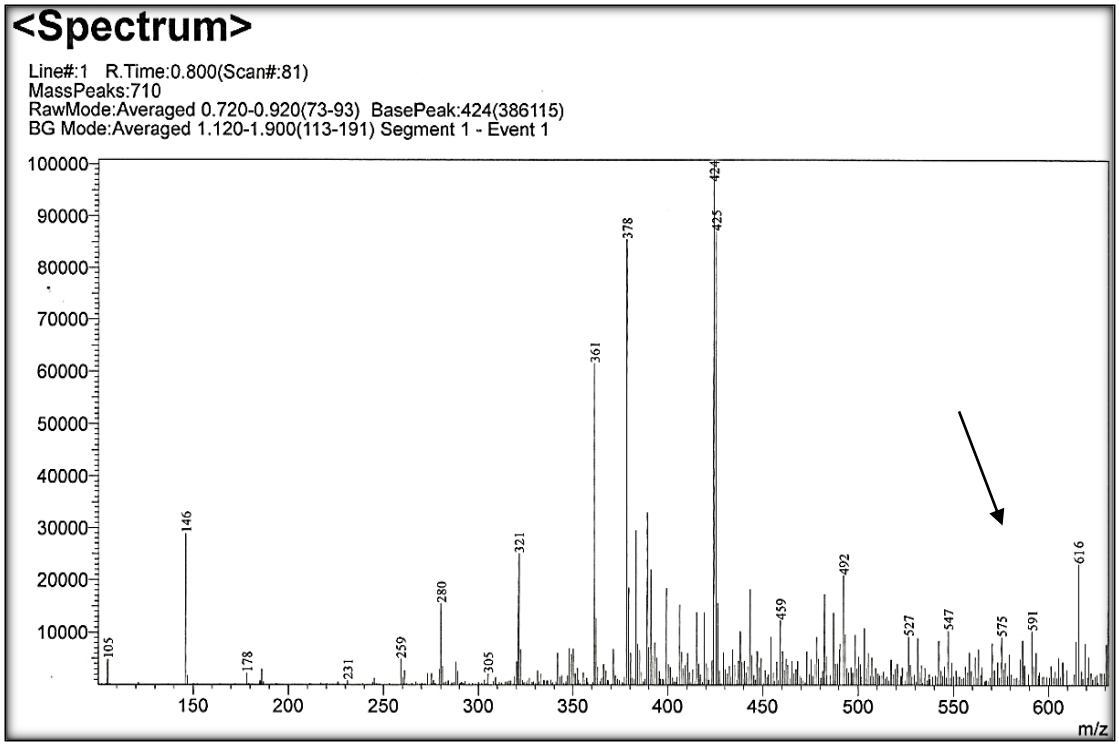


Fig.1. MS spectrum of Azo-Schiff base (I)

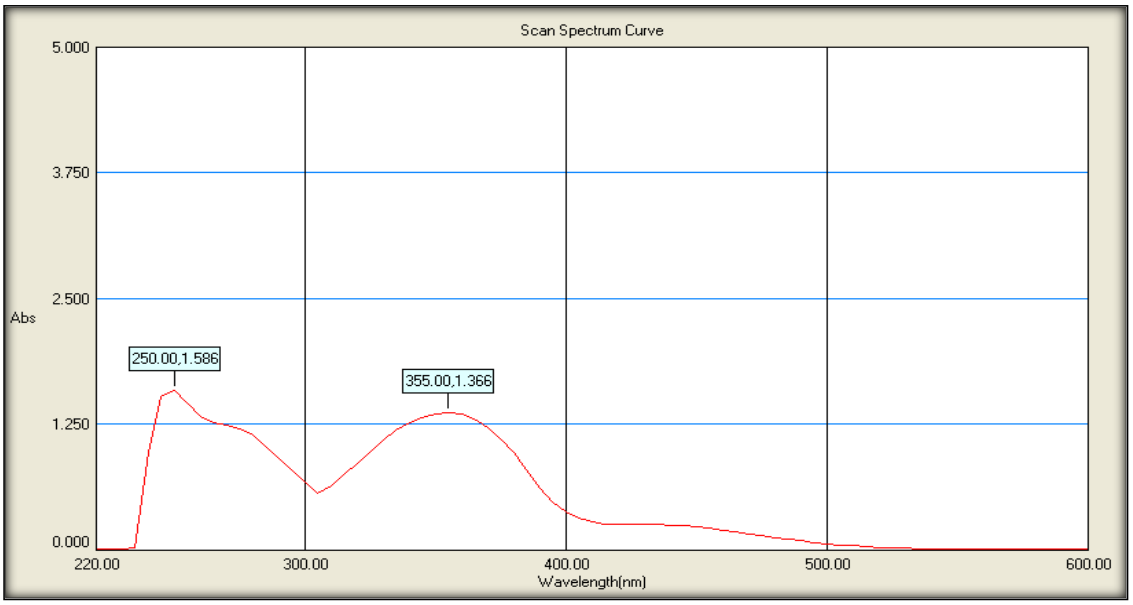


Fig.2. UV spectrum of Azo Schiff base (I)

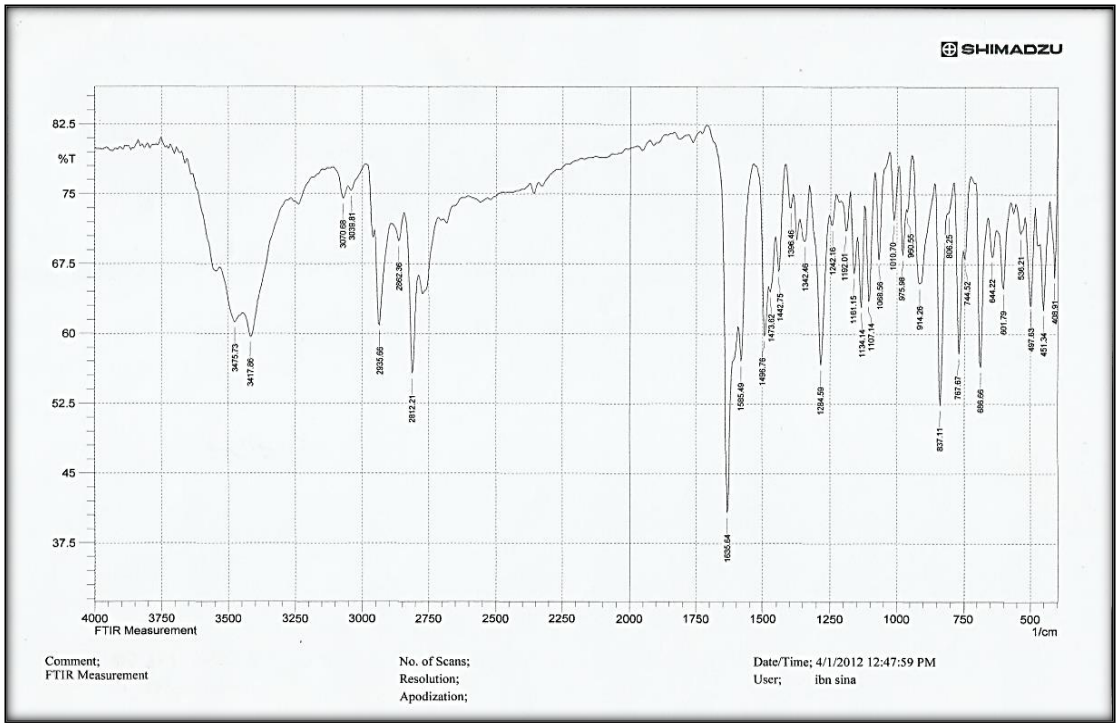


Fig.3. IR spectrum of Azo-Schiff ba se(I)

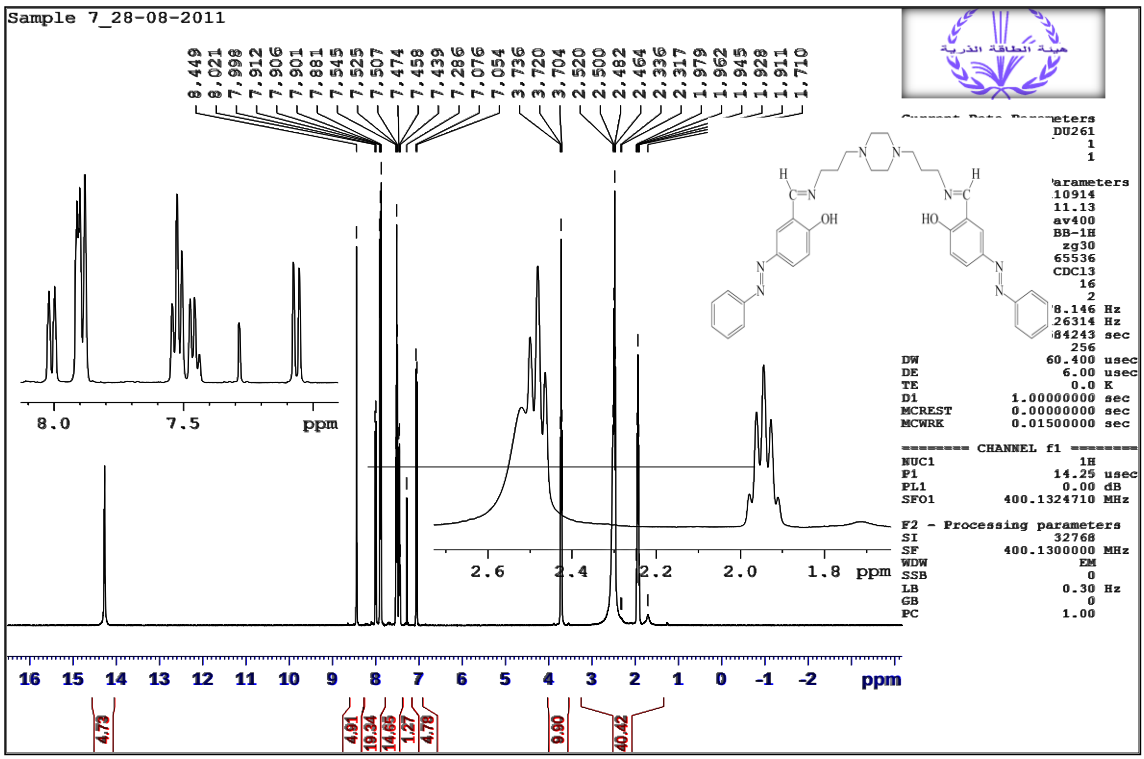
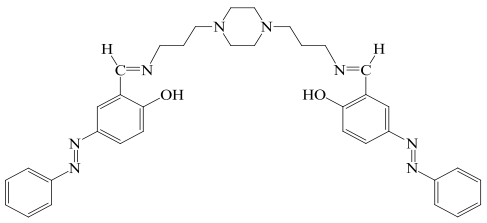
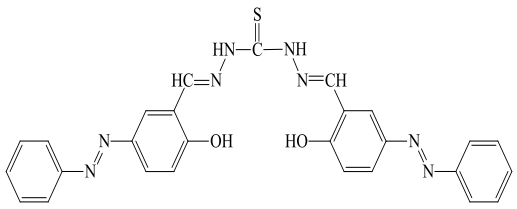


Fig.4. 1HNMR spectrum of Azo-Schiff base (I)

for 2 hour with continue stirrer. A solid mass formatted on hot. The reaction mixture was allowed to cool room temperature, it was suction filtered and recrystallized from suitable solvent, then dried in vacuum [36, 37] Yellow; Yield: (75 %), m.p=238^{0}C. Empirical formula :($C_{27}H_{22}N_8O_2S$).
M.Wt:(522 g).
Mass spectrum (m/z):522 (Fig.5.).
UV-Vis: λ_{max}= 355 nm (Fig.6.).
IR (KBr disk): 3421.72 -3448.72 cm^{-1} (O-H), 3205.69 cm^{-1} (N-H, amide), 3005.10cm^{-1} (C-H), aromatic), 1616.35 cm^{-1} (C=N), 1527.62-1558.48cm^{-1} (C=C, aromatic), 1442.75 – 1481.33 cm^{-1} (N=N), 1284.59 cm^{-1} (C-O), phenolic) (Fig.7.).
^{1}H-NMR ($CDCl_3$-400MHz) δ =12.311 (s, 2H, OH), 10.991 (s, 2H, N-H), 8.952 (s, 2H, CH=N), 7.125 -8.758 (m, 16 H, Ar), 2.509 - 3.387 (Organic solvents, DMSO, H_2O) (Fig.8.).
Elemental analysis: $C_{31}H_{23}N_7O_2$, Calculated (%) C: 62.06; H: 4.24; N: 21.44; O: 6.12; S, 6.14. Found (%) C: 62.11, H: 4.33, N: 21.25, O: 6.17, S: 6.14.



Scheme 2 Azo Schiff base (I)



Scheme 3 Azo Schiff base (II)

2.3. BIOLOGICAL ACTIVITY

The prepared compounds were tested for their antimicrobial activity against four species of bacteria (Bacillus subtilis, Escherichia coli , Staphylococcus aureus, Salmonella typhi) using filter paper disc method [38]. The screened compounds were dissolved individually in DMSO (dimethyl sulfoxide) in order to make up a solution of 50, 100, and 200 µg/ml concentration for each of these compounds. Filter paper discs (Whatman No.1 filter paper, 5mm diameter) were saturated with the solution of these compounds. The discs were placed on the surface of solidified Nutrient agar dishes seeded by the tested bacteria. The diameters of inhibition zones (mm) were measured at the end of an incubation period, which was 24 h at 37^{0}C for bacteria. Discs saturated with DMSO are used as solvent control. Ciprofloxacin 100µg/ml was used as reference substance for bacteria [38].

3. RESULTS AND DISCUSSION

3.1. SYNTHESIS

The prepared organic compounds (I,II) were synthesized by the condensation of 3-formyl-4-hydroxy phenylazo benzene with 1,4-Bis(3-aminopropyl) piprazine, Thiocarbohydrazide in the molar ratio 1:2 in absolute ethanol. The reactions proceeded smoothly, producing the corresponding Azo Schiff bases in good yield. The ligands are soluble in common organic solvent but insoluble in water. The structures of the ligand were elucidated by elemental analyses, MS, FTIR, electronic absorption, and 1H NMR spectra, which help in elucidating their empirical formula (Table 1).

3.2. ELEMENTAL ANALYSES OF AZO SCHIFF BASES

The results of elemental analyses Azo Schiff bases are in good agreement with those required by the proposed formula.

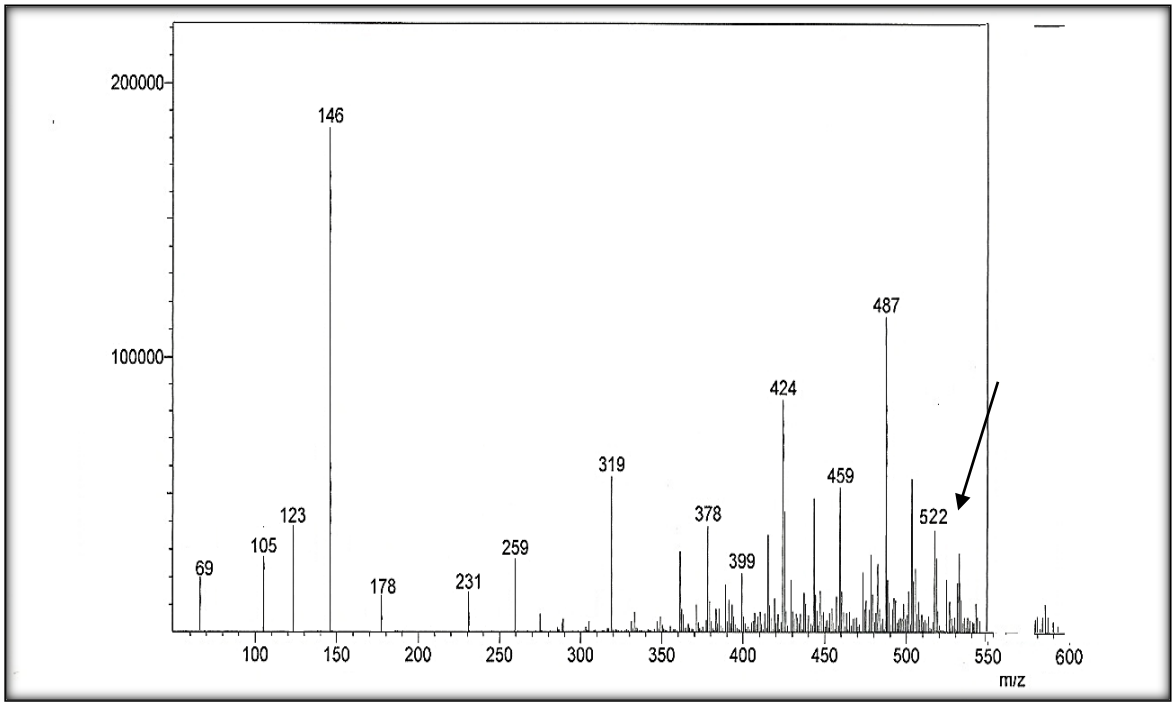


Fig.5. MS spectrum of Azo-Schiff base (II)

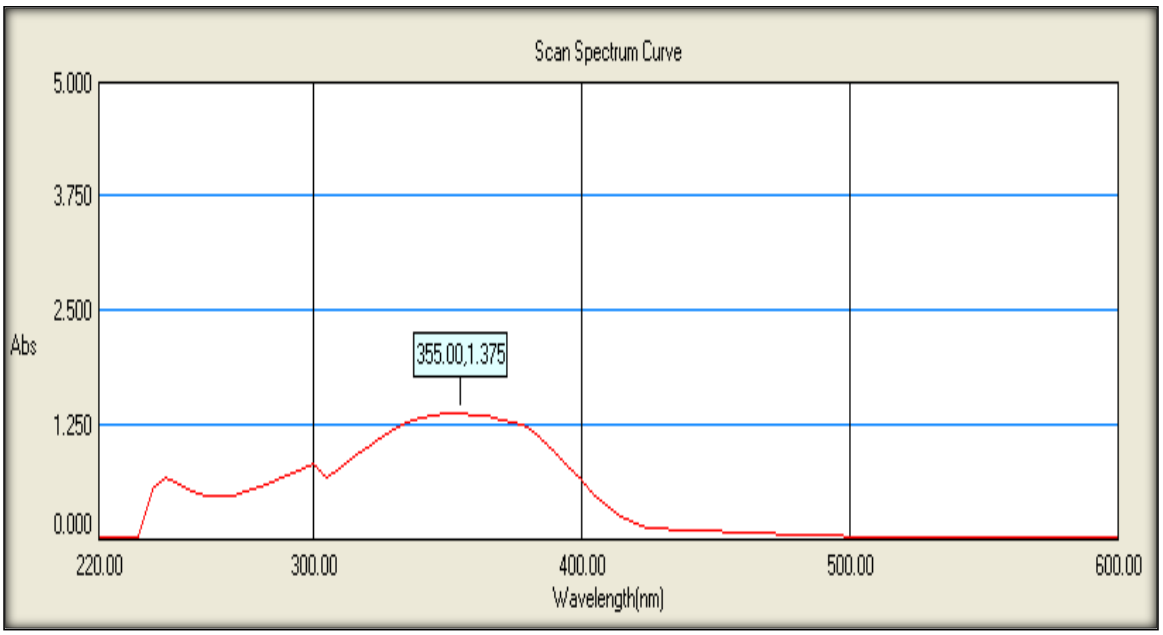


Fig.6. UV spectrum of Azo Schiff base (II)

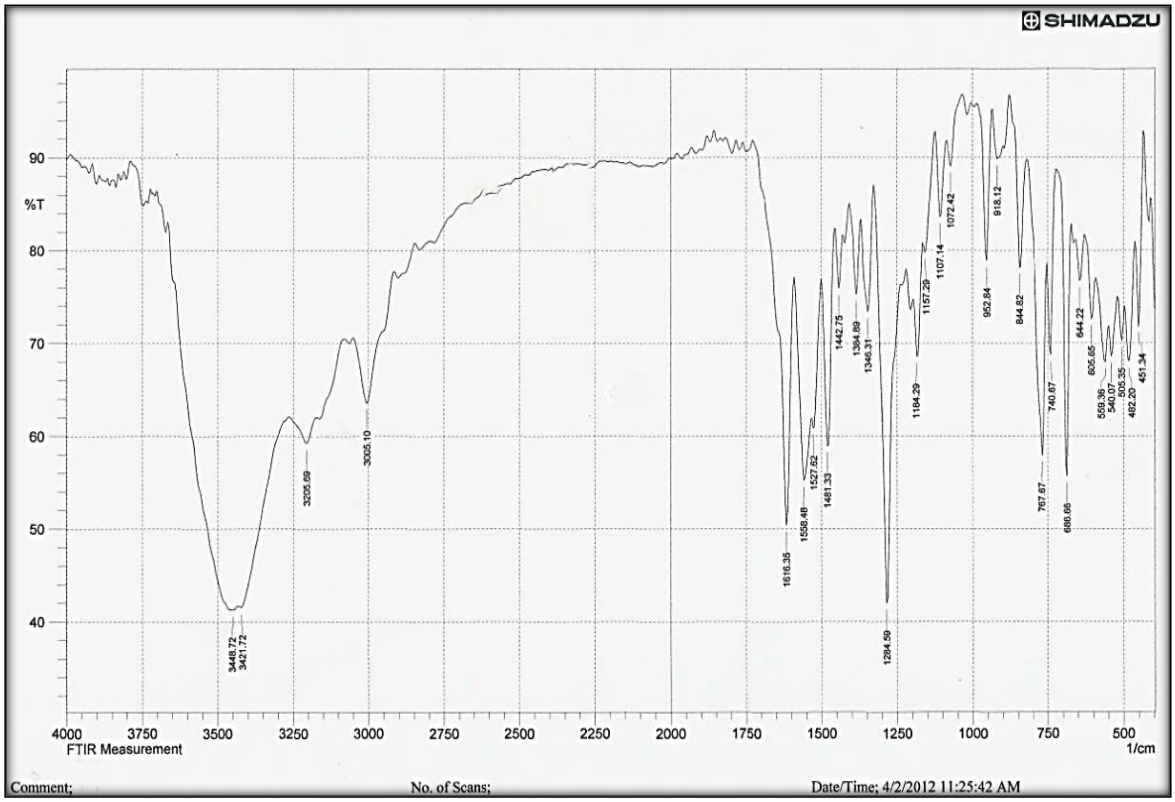


Fig.7. IR spectrum of Azo-Schiff base (II)

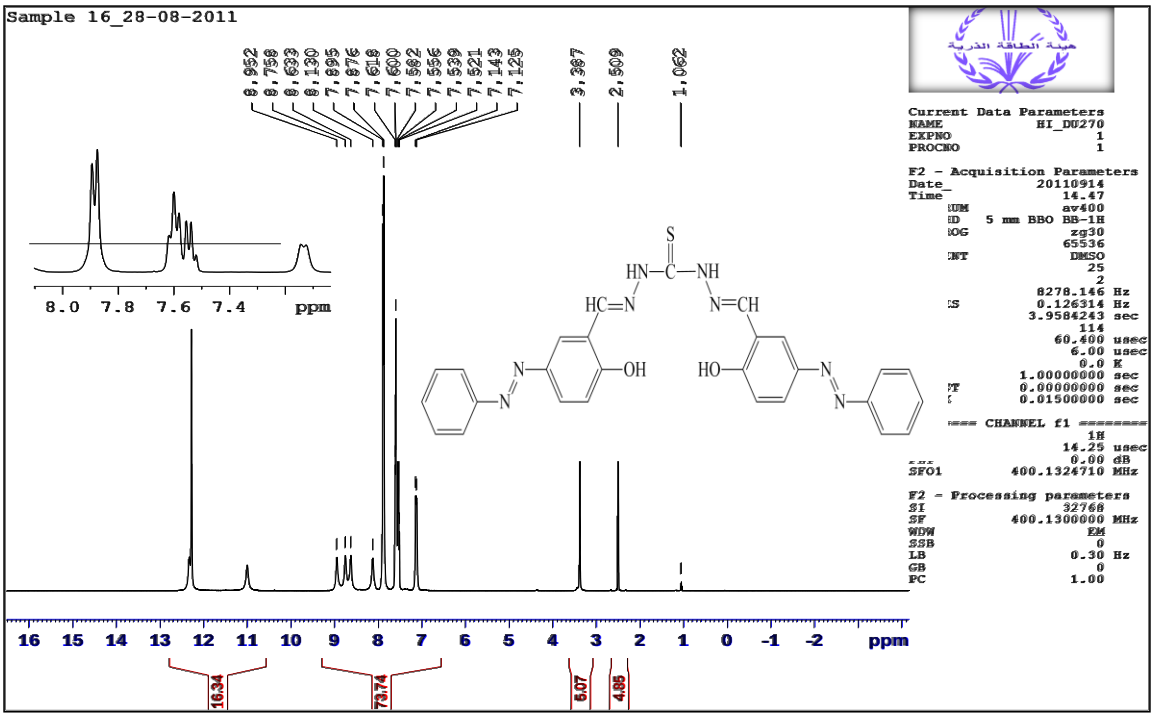


Fig.8. 1HNMR spectrum of Azo-Schiff base (II)

TABLE 1: COLOR, MOLECULAR WEIGHT, MELTING POINT OF (I, II).

AzoShiff base	Color	M.Wt	Melting point °C	λ_{max} nm	Yield %	Crystallization Solvent
I	Orange	616	227-228	355	75 %	Dioxan
II	Yellow	522	238	335	75 %	DMF &Water (9 : 1)

3.3. IR SPECTRA

In the IR spectra the five bands at 1635.64, 1616.35cm^{-1} are attributed to imine group (-HC=N-) for (I, II) (Fig 3 and 7), respectively. The bands in the spectra at 1585.49, 1527.62 and 1558.48 are due to (C=C) of aromatic rings. The ν(OH) stretching frequencies bands are observed at 3417.86-3475.73 and 3421.72 -3448.72 cm^{-1} for (I,II), respectively. The IR spectra of (I, II) shows characteristic absorption bands at 1473.62-1496.76 and 1442.75–1481.33 cm^{-1} due to ν(N=N) stretching vibrations, respectively. The bands which observed for all compounds at 1284.59 and 1284.59 cm^{-1} due to ν(C-O) vibration, while the band at 2812.21-2935.66 cm^{-1} are attributed to (C-H alkanes) for (I). Also, the bands at 3417.86-3475.73, and 3421.72 -3448.72 cm^{-1} are attributed to (C-H ar) for (I, II) respectively. However, in the IR spectra of Azo schiff bases this band (C=O) disappears and a new vibration band for azo methane (-HC=N-) is observed at 1635.64, and 1616.35 cm^{-1}, indicating that complete condensation takes place [39-40].

3.4. 1H NMR SPECTRA OF LIGANDS (I, II)

The 1H NMR spectra of (I, II) ligands in DMSO (Fig 4 and 8) shows a singlet signal at 14.28, and 12.311 ppm assigned to the protons of OH groups of the ligands (I, II) respectively, while the singlet signal at 10.991 ppm assigned to the protons of (CO-NH-) group of the ligand (II). The multiple signals 8.021 - 7.054 and 8.758 - 7.125 ppm are due to the aromatic protons for (I,II) respectively. The singlet signals at 8.449 and 8.952 ppm are due to the 2H, (HC=N) protons of the ligand (I,II) respectively.

The other obtained values for ^{1}H NMR chemical shifts of these compounds are given in the experimental section. These data are in agreement with those previously reported for similar compounds. These results strongly suggest that the proposed compounds have been formed [39-40].

3.5. MASS SPECTRA OF LIGANDS (I,II)

The compound (I) show peaks at 616 and many fragments at: 146, 176, 259, 321, 361, 492 and 575. Compound (II) show peak at 522 and many fragments at: 69, 105, 123, 146, 178, 399, 424, 487, 522 respectively.

3.6. BIOLOGICAL ACTIVITY

During the last two or three decades, attention has been increasing due to the synthesis of Azo Schiff bases which exhibits various biological activities including antibacterial, fungicidal, tuberculostatic and plant growth regulative properties [41]. It was judicious to investigate the synthesis of various new types of Azo Schiff bases and studied their antibacterial activity against four strains of bacteria (Bacillus subtilis, Escherichia coli, Staphylococcus aureus, and Salmonella typhi) (Fig. 9.).

The concentrations used for the screened compounds are 50, 100, and 200 µg/ml. Ciprofloxacin was used as reference standard while DMSO as control and inhibition zones are measured in mm. The new compounds were tested against one strain each of a gram positive and two gram negative. The test results are presented in Table 2.

All compounds were not active when used in the range between 50, 100 µg/ml but were active in the concentrations 200 µg/ml.

TABLE 2: EFFECT OF NEW AZO SCHIFF BASES ON THE GROWTH OF TESTED BACTERIA (CONC.200 MG/ML)

AzoShiff base	Bacteria			
	Gram negative		Gram positive	
	B. subtilis	*S. aureus*	*E.coli*	*S. typhi*
I	19mm	16 mm	15 mm	18 mm
II	20 mm	18 mm	16 mm	14 mm
Control	00 mm	00 mm	00 mm	00 mm
Ciprofloxacin	20 mm	20 mm	20 mm	20 mm

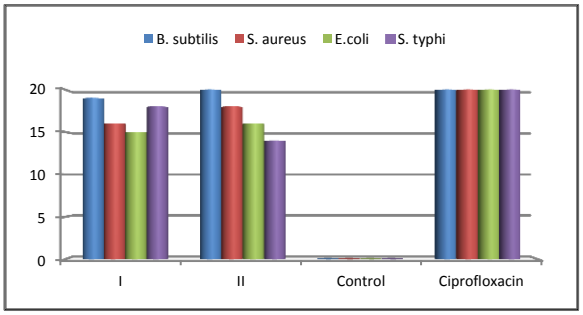


Fig.9. Antibactial activity (Gram positive, negative) of synthesized compounds

4. THERMAL STUDIES

Thermogravimetric (TG) analyses for the ligands were carried out within the temperature range from ambient temperature up to 900°C. The thermal behaviour of the ligands show good agreement with the formula suggested from the analytical data in table 3. The ligands (I, II) undergo two step decomposition processes (Fig. 10.). The first step of ligand (I) was started at 197°C to 210 °C show mass loss 2.283% (calcd. 2.922%), was attributed to the loss of water moiety while the first step of ligand (II) was started at 220°C to 330°C show mass loss 35.941% (calcd. 35.809%), This was attributed to the loss of $[C_6H_8N_4O.2H_2O]$ moiety. The second step of ligand (I) was started at 300°C to 450°C show mass loss 28.203% (calcd. 28.085%), This was attributed to the loss of $[C_7H_{11}N_5).H_2O]$ moiety. The second step of ligand (II) was started at 390°C to 665°C show mass loss 68.006% (calcd. 68 %). This was attributed to the loss of $[C_{24}H_{11}N_3O]$ moiety. Also the third step of ligand (I) was started at 450°C to 730°C show mass loss 72.24% (calcd. 71.1%). This was attributed to the loss of $-[C_{28}H_9N_3O_2]$ moiety.

5. KINETIC STUDY

The kinetic analysis parameters such as activation energy (ΔE*), enthalpy of activation (ΔH*), entropy of activation (ΔS*), free energy change of decomposition (ΔG*) were evaluated graphically by employing the Coats-Redfern relation [42].

$$\log [-\log (1- \alpha)/T2] = \log [AR/\theta E^*(1-2RT/E^*] - E^*/2.303RT \quad (1)$$

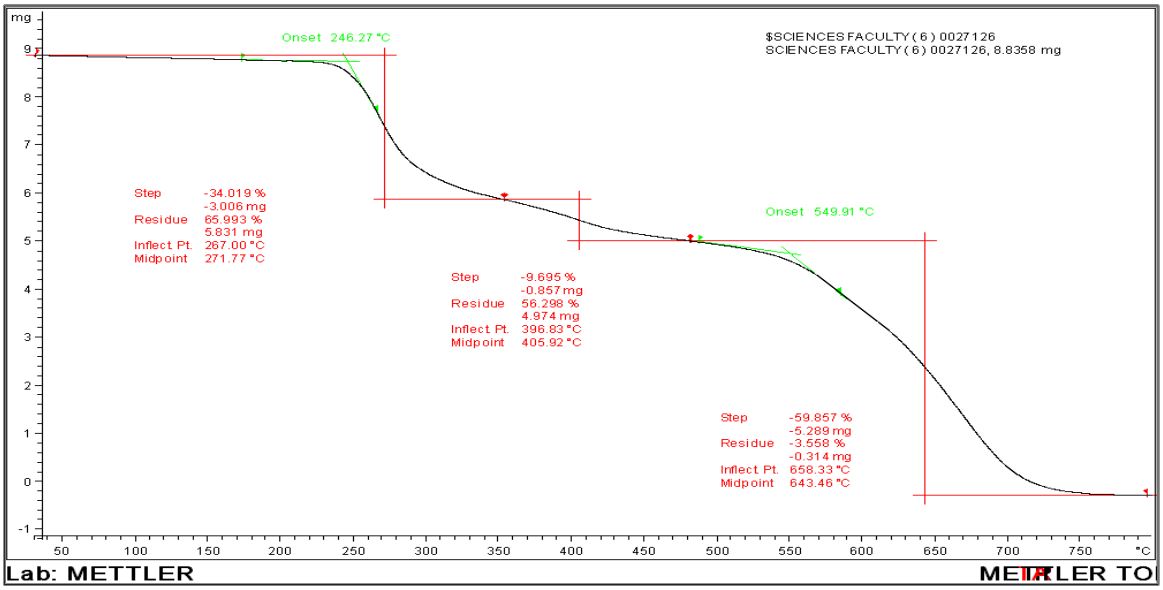
where α is the mass loss up to the temperature T, R the gas constant, E* the activation energy in $Jmol^{-1}$, θ the linear heating rate and (1-2RT/E*) ≈ 1. A plot of left hand side of Eq. (1) against 1/T gives aslope from which E* was calculated and A (Arrhenius constant) was determined from the intercept.

From relevant data, linearization plots (Figure 11-13) confirm first order kinetics. The entropy of activation (ΔS*) and the free energy change of activation (ΔG*) were calculated using equations (2) and (3):

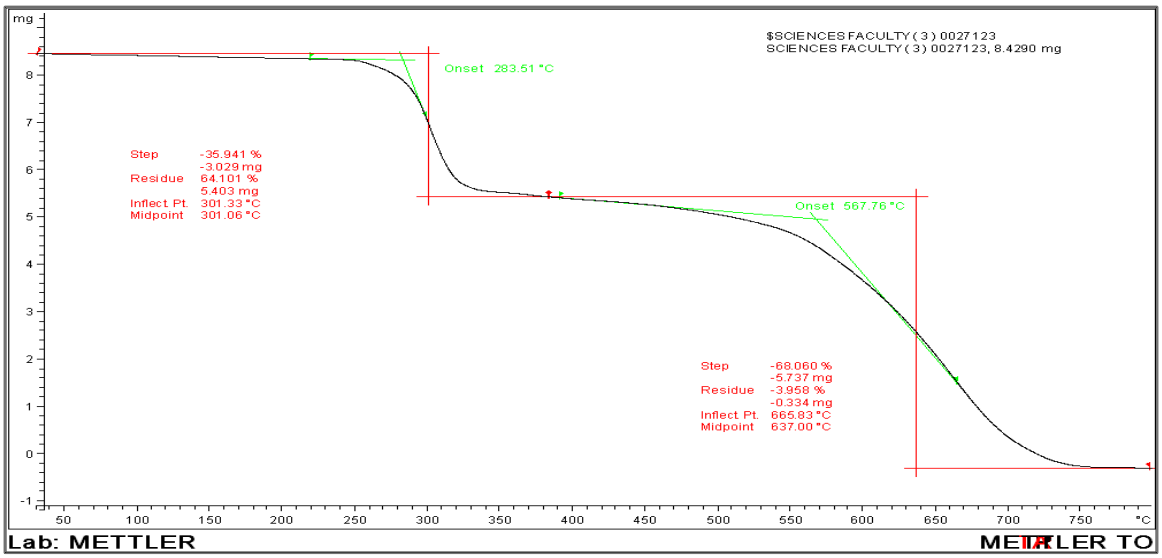
$$\Delta S^* (JK\text{-}1mol\text{-}1) = 2.303R [\log (Ah/kT) \quad (2)$$

$$\Delta G^*(Jmol\text{-}1) = \Delta H^* - T\Delta S^* \quad (3)$$

where k and h are the Boltzman and Plank constant, respectively. The calculated values of ΔE*, A, ΔS* and ΔG* for the decomposition steps of the ligands are given in Table 4. According to the kinetic data obtained from the TG curves, all the ligands



(I)



(II)

Fig.10. TG curve of ligand (I) and (II)

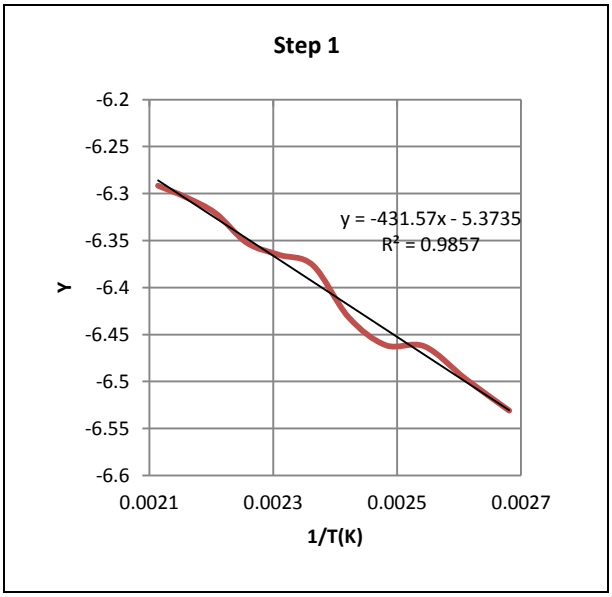
TABLE 3: THERMO ANALYTICAL DATA OF AZO-SHIFF BASES

Ligands	steps	T_i ^0C	T_f ^0C	T_{max} ^0C	Δm% Found (cal.)	Assignment
I	1	197	210	207.83	2.283 (2.922)	-[H_2O]
	2	300	450	377.02	28.203 (28.085)	-[$C_7H_{11}N_5$).H_2O
	3	450	730	646.60	72.24% (71.1)	[$C_{28}H_9N_3O_2$]
	final residue:(found: 2.702%), [Calc (2.597%)].					
II	1	220	330	326.92	35.941 (35.809)	- [$C_6H_8N_4O.2H_2O$]
	2	365	700	460.18	68.006 (68)	- [$C_{24}H_{11}N_3O$]
	final residue:(found: 3.958 %), [Calc (3.834%)].					

TABLE 4: THERMODYNAMIC ACTIVATION PARAMETERS OFÂZO-SHIFF BASES

Ligands	Order n	steps	R^2	A s^{-1}	E_a $kJ.mol^{-1}$	ΔS^* $J.K^{-1}mol^{-1}$	ΔH^* $kJ.mol^{-1}$	ΔG^* $kJ.mol^{-1}$
I	1	1	0.985	$1.5x10^{-4}$	23.71	-321.9	19.72	174.52
	1	2	0.989	258.69	98.17	-205.2	92.77	226.16
	1	3	0.999	$3.7x10^{-6}$	-10.14	-358.3	-17.78	311.69
II	1	1	0.96	17.27	72.19	-226.4	67.57	193.57
	1	2	0.99	$3.3\ x10^{-6}$	-10.99	-358.4	-17.97	283.36

have negative entropy, which indicates that the ligands are formed spontaneously. The values of the activation energy in the Table 4, we find that the activation energy of the second step of the disintegration associated with (I) is larger than activation energies for the first step, and this indicates that the rate of decomposition in the second step is less than the rate of decomposition in the first step. This is due to residual hardness of ligand structure, which requires a higher power to restore order and organization before any change in the composition and vice versa As the negative values of the energies of activation is energy-extensive the resistance solid inside the body that lead to the spread of heat [43]. Also we find that the positive values for (ΔG^*) to indicate that the non-spontaneous reactions when changing situation. The negative values for (ΔS^*) indicate that the union has more ordered structure of the reactants and the reactions are slow more than the natural or formal. While positive values for (ΔH^*) s indicate that the endothermic reactions. And negative values indicate that the exothermic reactions and small values for (Arrhenius constant)(A) shows that the dissociation reactions for quick ligand while large and positive values when the case of the transition can be classified as slow reactions [44].



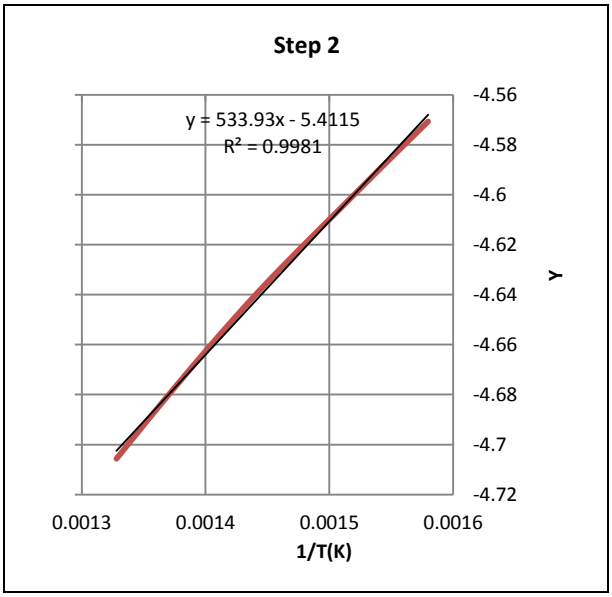


Fig.11. Linearization plot of ligand (I) when $Y = \log\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right]$ for $n \neq 1$

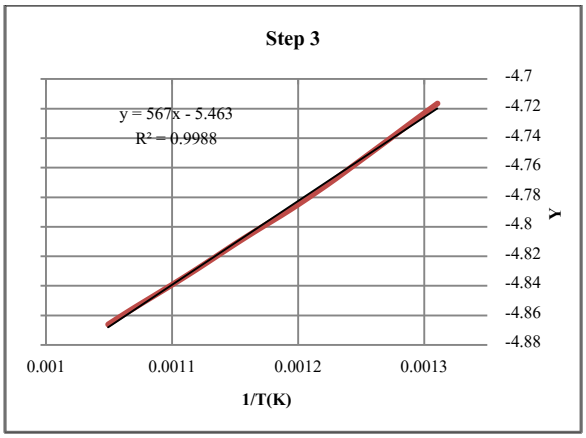
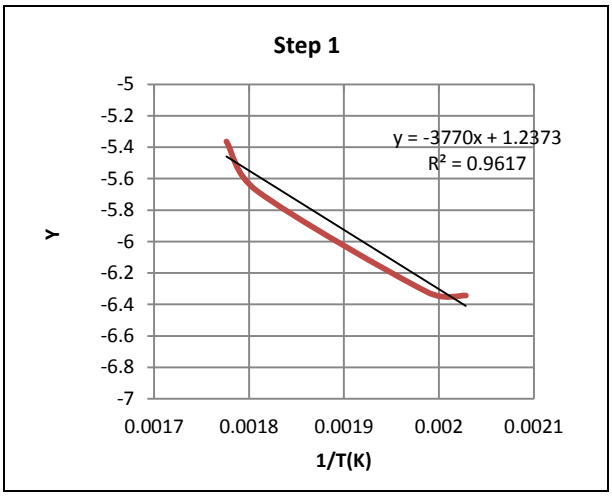


Fig.12. Linearization plot of ligand (I) when $Y = \log\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right]$ for $n \neq 1$



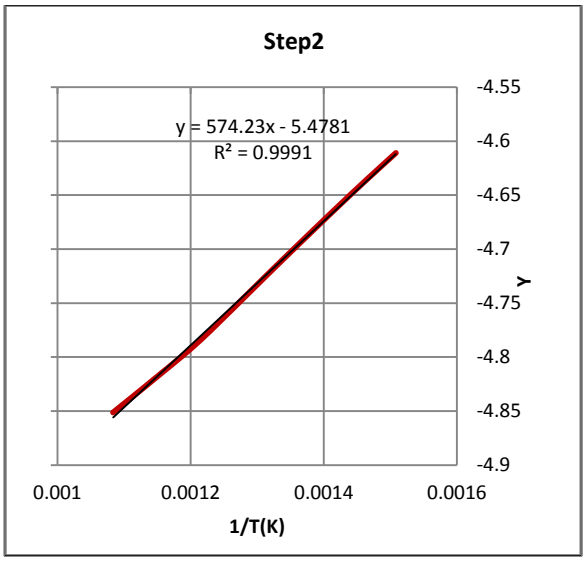


Fig.13. Linearization plot of ligand (II) when $Y = \log\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right]$ for $n \neq 1$

6. CONCLUSION

1- The (I, II) compounds are new and were prepared for the first time.

2- The new compounds were identified by 1HNMR, IR, LC-MS spectral methods.

3-Some of the prepared compounds have been biologically screened i.e. studying their effects against two gram-positive, two gram-negative bacteria. The results show that their activities were found to vary from moderate to very strong.

4- The thermal degradation behavior and thermal stability of the ligands (I, II) are discussed. The thermodynamic parameters of decomposition processes of Ligands, namely, activation energy (Eα), enthalpy change (ΔH*), entropy change (ΔS*), and Gibbs free energy change (ΔG*) were evaluated graphically by employing the Coats-Redfern method.

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تحضير ودراسات طيفية , حركية, التحلل الحرارى و مضادات الميكروبات لقواعد الازوشيف

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الملخص:

في هذا البحث تم التركيزعلى تحضير وتمييز بعض قواعد الأزو شيف والمحضرة من تكثيف ٣ –فورميل ٤- هيدروكسى فينيليزو البنزين مع كلا من ٤,١ باى (٣أمينو بروبيل) بيبرازين و ثيو كاربوهيدرازيد على التوالي. تم دراسه قواعد الأزو شيف (LC-MS، GC-MS، 1H- NMR، IR) باستخدام تقنية الأطياف تم دراسه نشاط قواعد الأزو شيف كمواد مضاده للجراثيم (العضوية الرقيقة والمكورات العنقودية الذهبية) واثبت الدراسه انها موجبه الجرام وأن (السالمونيلا التيفية وكولاي) هي سلبية الجرام أظهرالبحث أن يجند الازو له نشاطا متغيرا قادرا على تثبيط نمو البكتيريا وقد تعرضت قواعد الأزو شيف للتحليل الحراري المتزامن وتم دراسه آلية التحلل والاستقرار الحراري.