



Synthesis, Fluorescence and Thermal Properties of Some Benzidine Schiff Base

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Abstract

A series of Schiff bases derived from benzidine and various aromatic aldehydes were prepared and characterized by spectroscopic methods. Fluorescence properties of prepared Schiff bases were studied in DMF solution. The thermo kinetic parameters. E , ΔH , ΔS and ΔG were calculated following Coats-Redfern method.

Key Words: Thermal Properties, Benzidine, DMF Solution.

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Introduction

Schiff bases benzidine are class of compounds in chemistry and have pharmacological, industrial, as well as many other applications as an intermediate in the syntheses of other compounds such as lactams (Hitoshi et al, 1997; Punniyamurthy et al, 1995; Trivedi and Desai, 1992) or metal complexes where Schiff bases consider a good ligand especially the Schiff bases derived from salicylaldehyde, hydroxy naphthaldehyde and their metal complexes consider a good catalysis in some organic reactions such as oxidation reaction, photoluminescence, electroluminescent devices and biological active compounds, the thermal stability and kinetic parameters were study by many other followed well known method of calculation one of the most used method is Coats-Redfern approximation which consider the most proper method to calculation the parameters with accurate close to 98% (Kumari et al, 2009; Leçe et al, 2008; Li et al, 2009; Pui et al, 2011; Shakya et al, 2011; Ziółek and Sobczak, 2008; Karakoç et al, 2019; Warad et al, 2020; Gesawat and Shakeel, 2017; Zoubi et al, 2018; Görgülü, 2018; Kalofolias et al, 2019).

In the study the Schiff bases bis (p-chlorobenzaldehyde, 3-methoxy -2- hydroxyl benzaldehyde, 2-hydroxy benzaldehyde, 2,4-dimethoxy benzaldehyde, phenanthrene-9-carbaldehyde and indol-3-carboxaldehyde) benzidine were prepared by standard methods and characterized by the determination of melting point. IR, H-NMR, MASS, Fluorescence spectral measurement and thermal analyses (TG -DTA).

Experimental

1. Materials

Benzidine, p-chlorobenzaldehyde, 2-hydroxyl-3-methoxybenzaldehyde, 2-hydroxybenzaldehyde, 2,4-di methoxy benzaldehyde, phenanthrene-9-carboxaldehyde and indol-3-carboxaldehyde, Methanol, glacial acetic acid, dimethylformamide (DMF), were supplied from Merck Chemical Co. (Germany).

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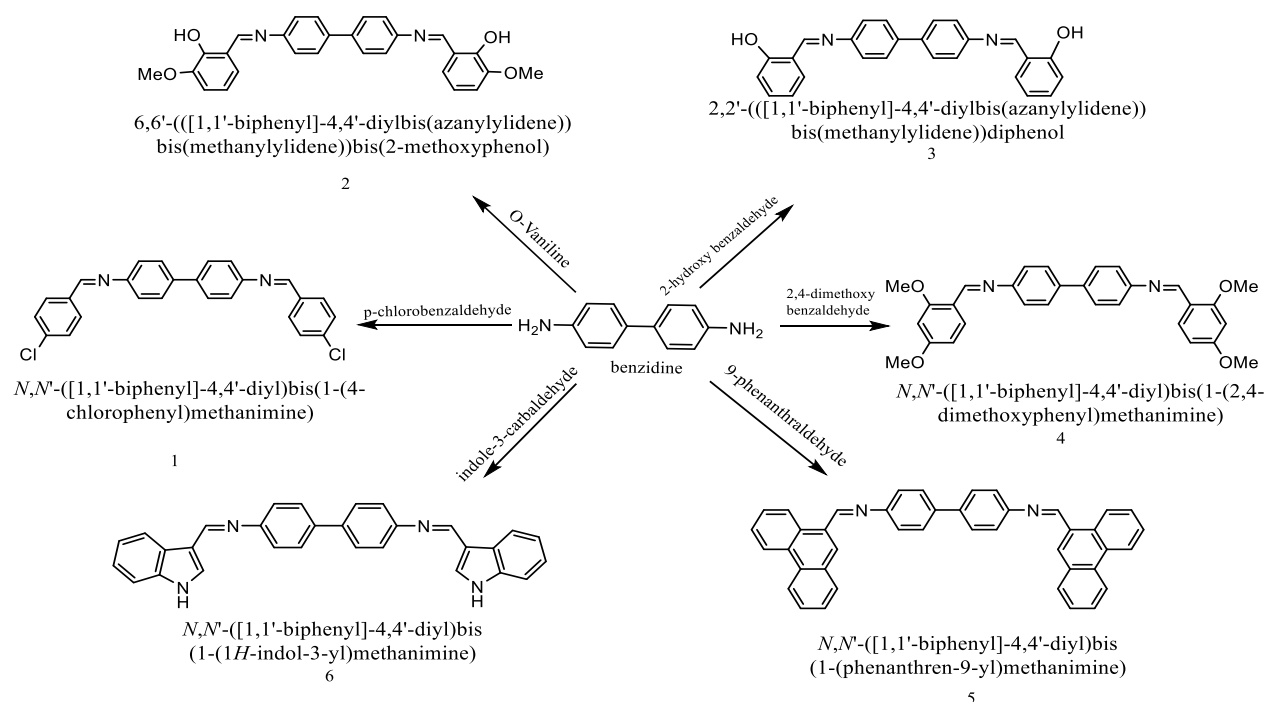


2. Instrumentation

Melting point was recorded using thermal scientific apparatus in open capillary tube. FT-TR spectra were recorded as KBr pellets on a Shimadzu FT-IR 8400S. Schiff bases samples were dissolved in dimethyl sulfoxide-*d*₆ to obtain ¹H-NMR spectra using (Bruker, 400 MHz), tetra methyl silane was used as the internal standard. EI-mass were recorded by Agilent Technologies 5973C spectrometer.

U-Vis and fluorescence spectra (in DMF) were recorded on Analytic Jena spectrophotometer and Cary Eclipse Spectrofluorophotometer. (TG-DTA) were recorded from room temperature to 600°C under N₂ with heating rate 10°C /min using PerkinElmer.

3. Preparation of the benzidine Schiff base (1, 2, 3, 4, 5 and 6)



Scheme 1. Synthesis Schiff base compound

Spectral and Analytical data of the synthesized benzidine Schiff bases were as a following:

***N, N'*-([1,1'-biphenyl]-4,4'-diyl) bis(1-(4-chlorophenyl) methanimine) (1)**

Yellow solid; Melting point: 268-269°C, Yield: 91%; FT- IR (KBr, cm⁻¹) spectra showed the following bands: 2877 (Ar-H); 1620 (C=N); 1489-1585 (C=C);

Schiff base 1, 2, 3, 4, 5 and 6 were synthesized by mixing of benzidine (2mmol, 0.368g) dissolved in methanol (15 mL) with p-chlorobenzaldehyde (4mmol, 0.56gm), o-vanillin (2-hydroxyl-3-Methoxy benzaldehyde), (4mmol, 0.60gm), 2-hydroxybenzaldehyde (4mmol, 0.50gm), 2,4-dimethoxybenzaldehyde (4mmol, 0.54gm), phenanthrene-9-carboxaldehyde (4 mmol, 0.824 g) and indol-3-carboxaldehyde (4mmol, 0.58gm) in methanol (15 mL) for each respectively. Drops of glacial acetic acid was added and was refluxed for (3-5) hours, The resulting solids which obtain during the refluxing process were filtered hot and wash with cold methanol and dried in vacuum. (Santosh et al, 2010; Anuradha and Rajavel, 2011). (Scheme 1) shows the synthesis of benzidine Schiff bases.

1091 (Ar -N); 837(C-Cl); λ_{flu} (DMF, max, nm): 422. λ_{exc} (DMF, max, nm): 342.

***6,6'*-((1,1'-biphenyl)-4,4'-diylbis(azanylylidene)) bis (methanylylidene)) bis (2-methoxyphenol) (2)**

Orang solid; Melting point: 190-191°C ; Yield: 91%; FT-IR (KBr, cm⁻¹): 3448 (OH); weak absorption bands at 2877-2982 (Ar-H, aliphatic -H); 1616 (C=N); 1454-1570 (C=C); 1249 (C-O-C); 1199 (C-O)

and (-C-N) appear at stretching frequency 1084; 1H-NMR (DMSO-*d*₆, δ / ppm): 13.42 (s, 2H, OH); 9.1 (s, 2H, Ar-CH=N); 7.6-7.8 (8H, m, Ar-H) referred to biphenyl, 6.98-7.3 (6H, m, Ar-H) due to substituted phenyl and (6H, s, Ar-OCH₃) for methoxy groups in meta position at δ = 4.14 ppm. λ_{flu} (DMF, max, nm): 422. λ_{exc} (DMF, max, nm): 342.

2,2'-([1,1'-biphenyl]-4,4'-diylbis (azanylylidene)) bis (methanylylidene)diphenol(3)

Yellow solid; Melting point: 261-262°C; Yield: 91%; FT- IR (KBr, cm⁻¹): 3448 (OH, phenol); 2989-3055 (Ar-H); 1620 (C=N); 1489- 1570 (C=C); 1280 (C-O); 1188 (-C-N), 1H-NMR (DMSO-*d*₆, δ / ppm): 13.32 (2H, s, OH); 9.0 (2H, s, Ar-CH=N); 7.72 and 7.62 (8H, m, Ar-H) referred to biphenyl; 6.93-7.27 (8H, m, Ar-H) referred to substituted phenyl. λ_{flu} (DMF, max, nm): 422. λ_{exc} (DMF, max, nm): 342

N,N'-([1,1'-biphenyl]-4,4'-diyl) bis (1-(2,4-dimethoxyphenyl) methanimine)(4)

Yellow solid; Melting point: 270-271°C; Yield: 93%; FT- IR (KBr, cm⁻¹): 3001-2839 (Ar-H, aliphatic-H); 1604 (C=N); 1269 (CH₃); 1161 (C-N); 1500- 1581 (C=C); 1H-NMR (DMSO-*d*₆, δ / ppm): 3.0 and 3.9 (12H, s, CH₃); 8.83 (2H, s, Ar-CH=N); 7.43- 8.03 (8H, m, Ar-H) referred to biphenyl; 7.26 and 7.34 (2H, m, Ar-H); 6.7 and 6.6 (4H, m, Ar-H) referred to substituted phenyl. λ_{flu} (DMF, max, nm): 422. λ_{exc} (DMF, max, nm): 342.

N,N'-([1,1'-biphenyl]-4,4'-diyl)bis(1-(phenanthren-9-yl)methanimine)(5)

Yellow solid; Melting point: 312-313°C; Yield: 89%; FT- IR (KBr, cm⁻¹): 3448 (Ar-CH=N); 1624 (C=N);

3066 and 3028 (Ar-H); 1577 and 1489 (Ar -rings); 1199 (-C-N); 1H-NMR (DMSO-*d*₆, δ / ppm): 7.52 (4H, t, JHH 7.9 Hz, Ar-H); 7.63-7.72 (6H, m, Ar-H); 7.97 (4H, dd, Ar-H); 8.23 (2H, m, Ar-H); 8.31 (2H, d, Ar-H); 8.61 (2H, d, Ar-H); 8.85 (2H, s, CH=N). λ_{flu} (DMF, max, nm): 422. λ_{exc} (DMF, max, nm): 342

N,N'-([1,1'-biphenyl]-4,4'-diyl)bis(1-(1H-indol-3-yl)methanimine).(6)

Yellow solid ; Melting point: 179-182 °C; Yield: 77%; FT- IR (KBr, cm⁻¹) : 3171(s, NH, Indole); 2820-3109 (Ar-H); 1639 (C=N); 1446 - 1577 (C=C); 1242-1392 (-C-N); 1H -NMR in DMSO-*d*₆, showed : 7.28-7.323 (10H, m, Ar-H); 8.361 (2H, d, J=7.5Hz, CH=N); 9.99 (1H s, indole H-2); at δ = 7.57 ppm (4H, dd, Ar-H) and 8.13-8.15 ppm (4H, m, Ar-H) referred to biphenyl.

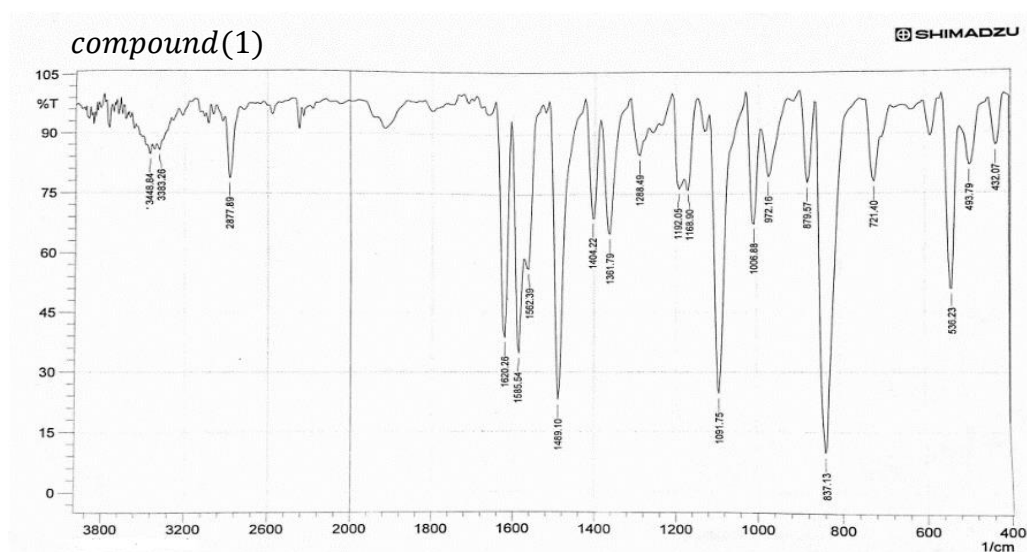
Results and Discussion

Spectroscopic Identification

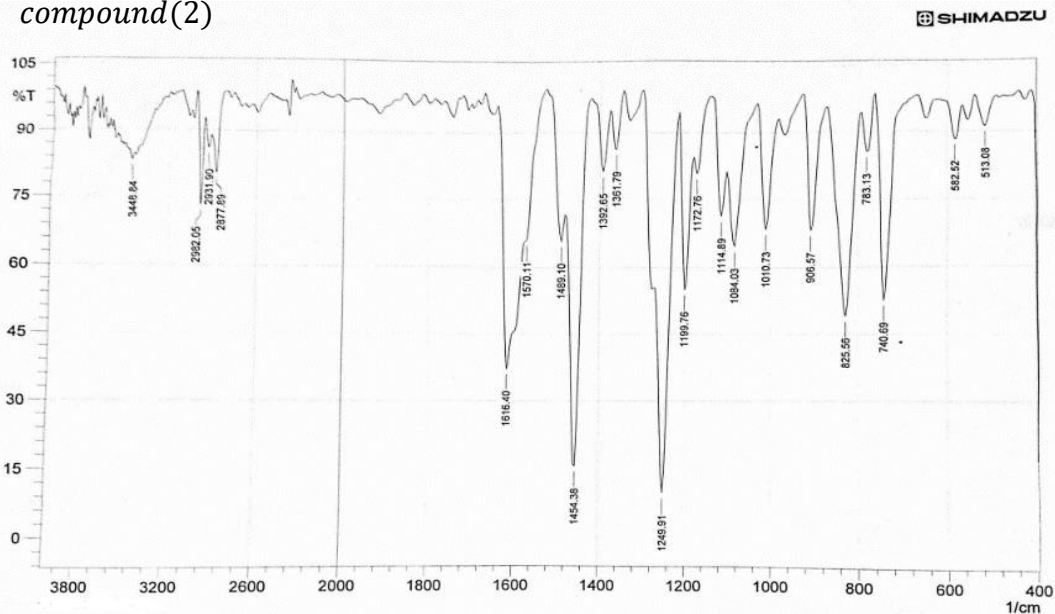
1. IR spectra

The condensation of benzidine with aromatic aldehyde were condensed in 1:2 molar ration giving very stable Schiff bases, the IR spectra of all compounds shows in Figure (1) a strong band in the range 1604-1624.12cm⁻¹. Which indicate the formation of Schiff bases and the totally absence of carbonyl stretching of aldehyde indicate the condensation occurs on the one and four position of benzidine. The other group in each compound show the expected band in the expected position as cited in the experimental part. (Anuradha and Rajavel, 2011; Wahba et al, 2017).

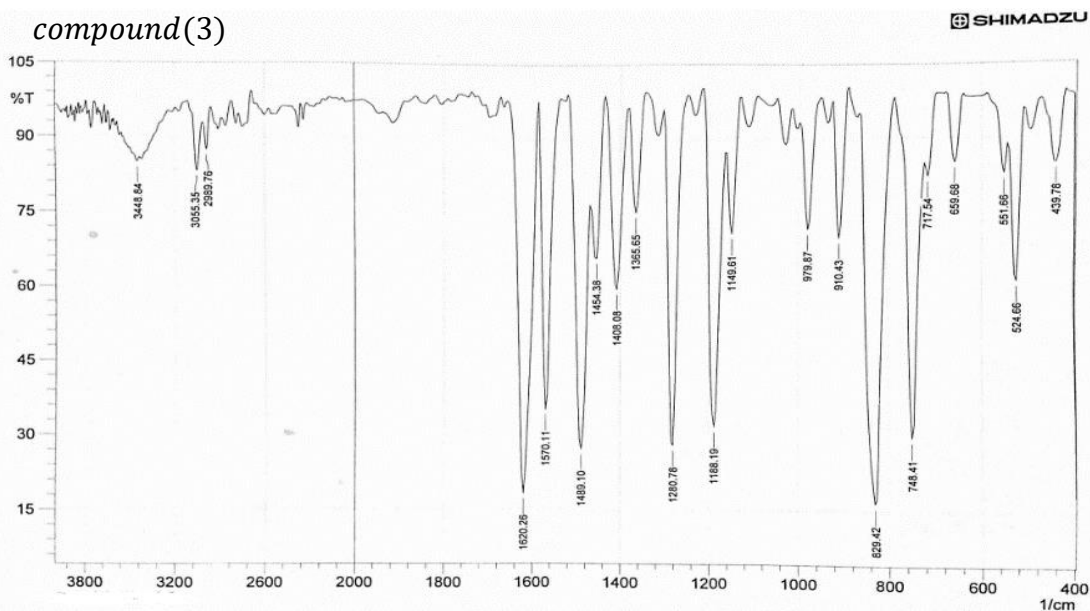
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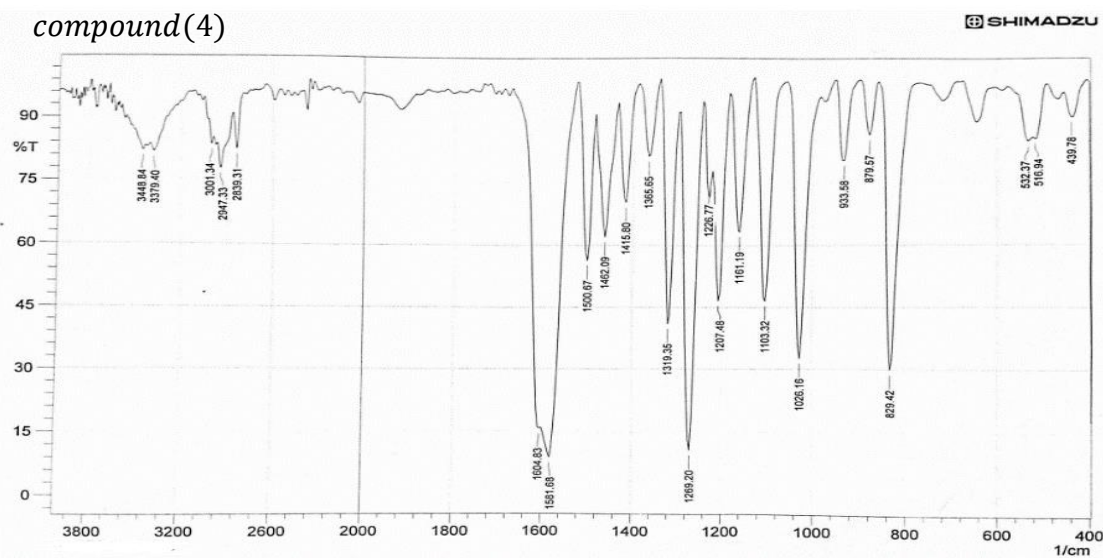
compound(2)



compound(3)



compound(4)



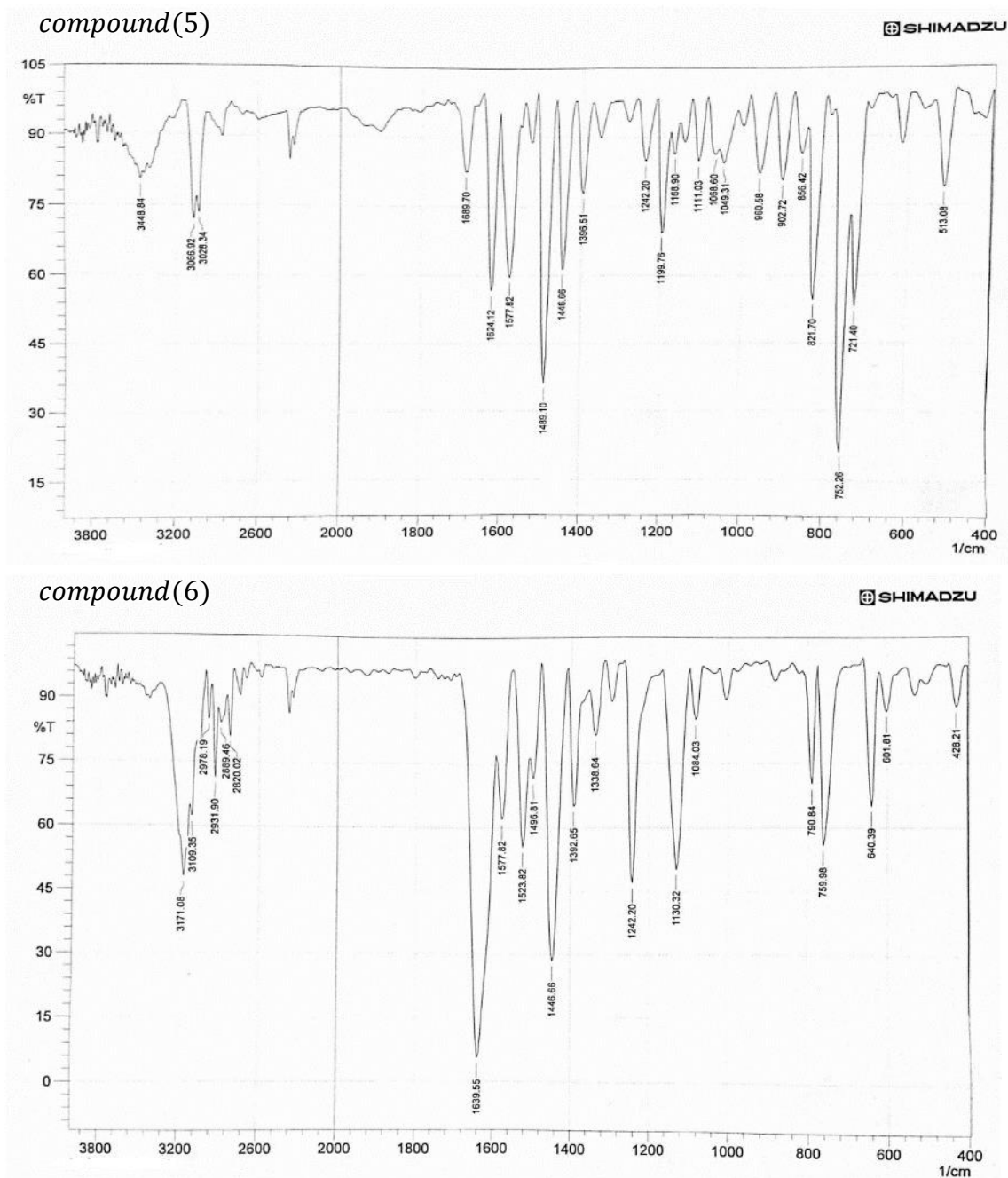


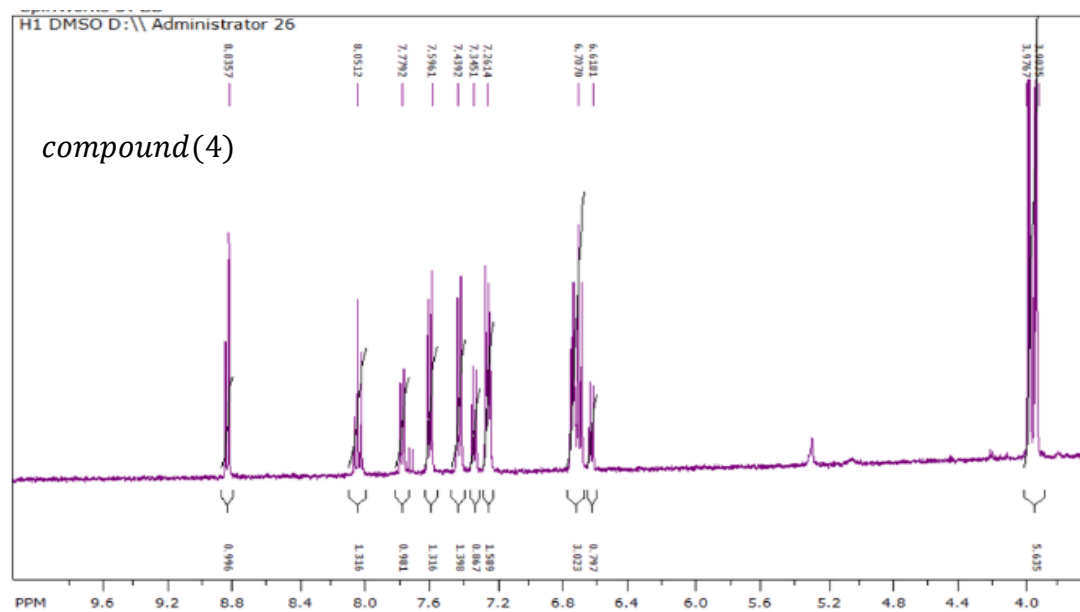
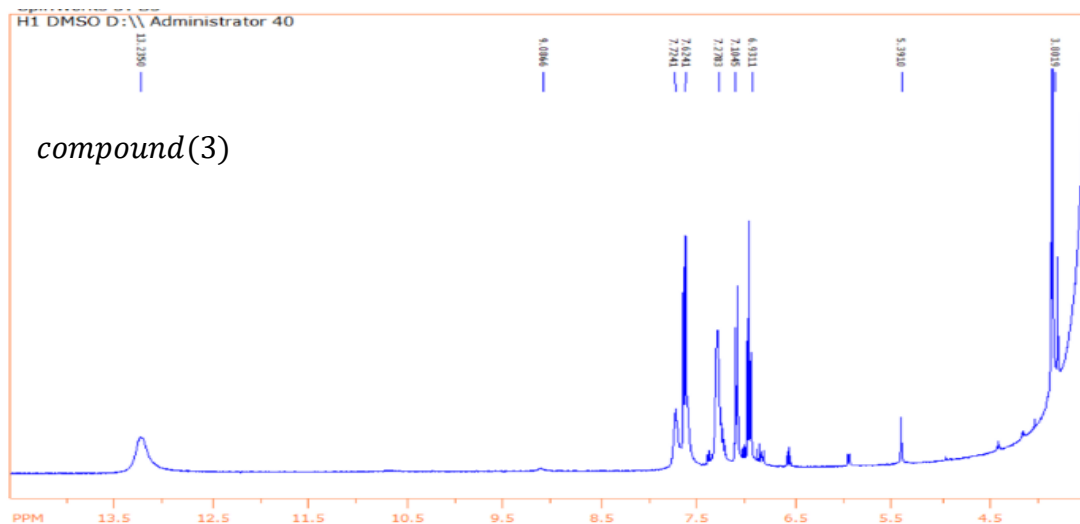
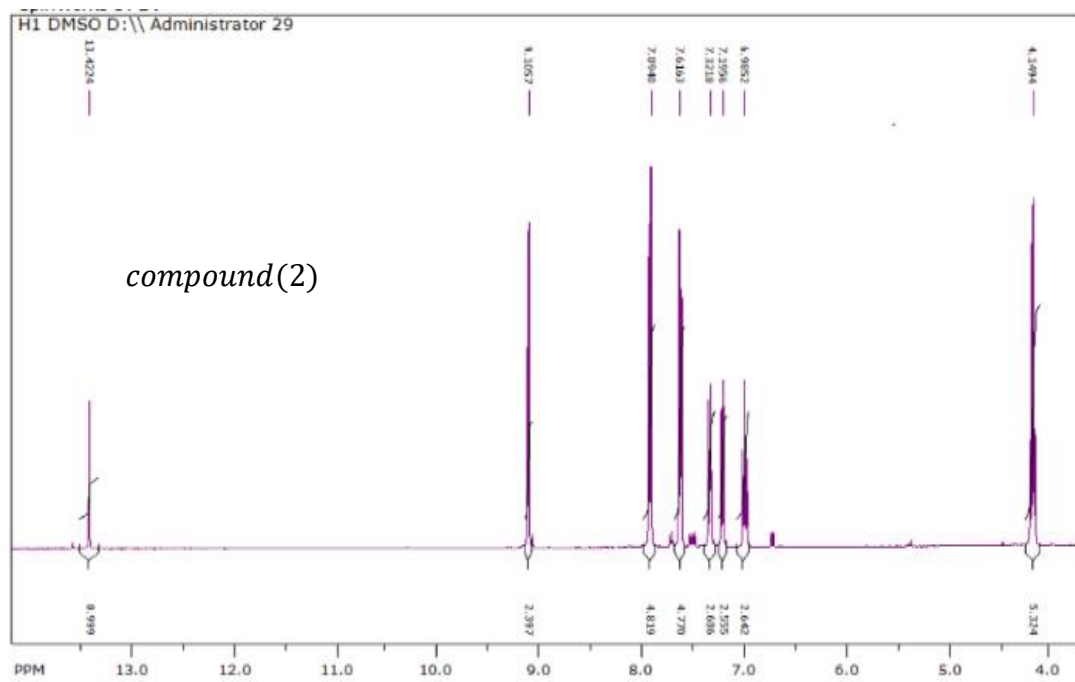
Figure 1. IR-spectra of compounds (1-6)

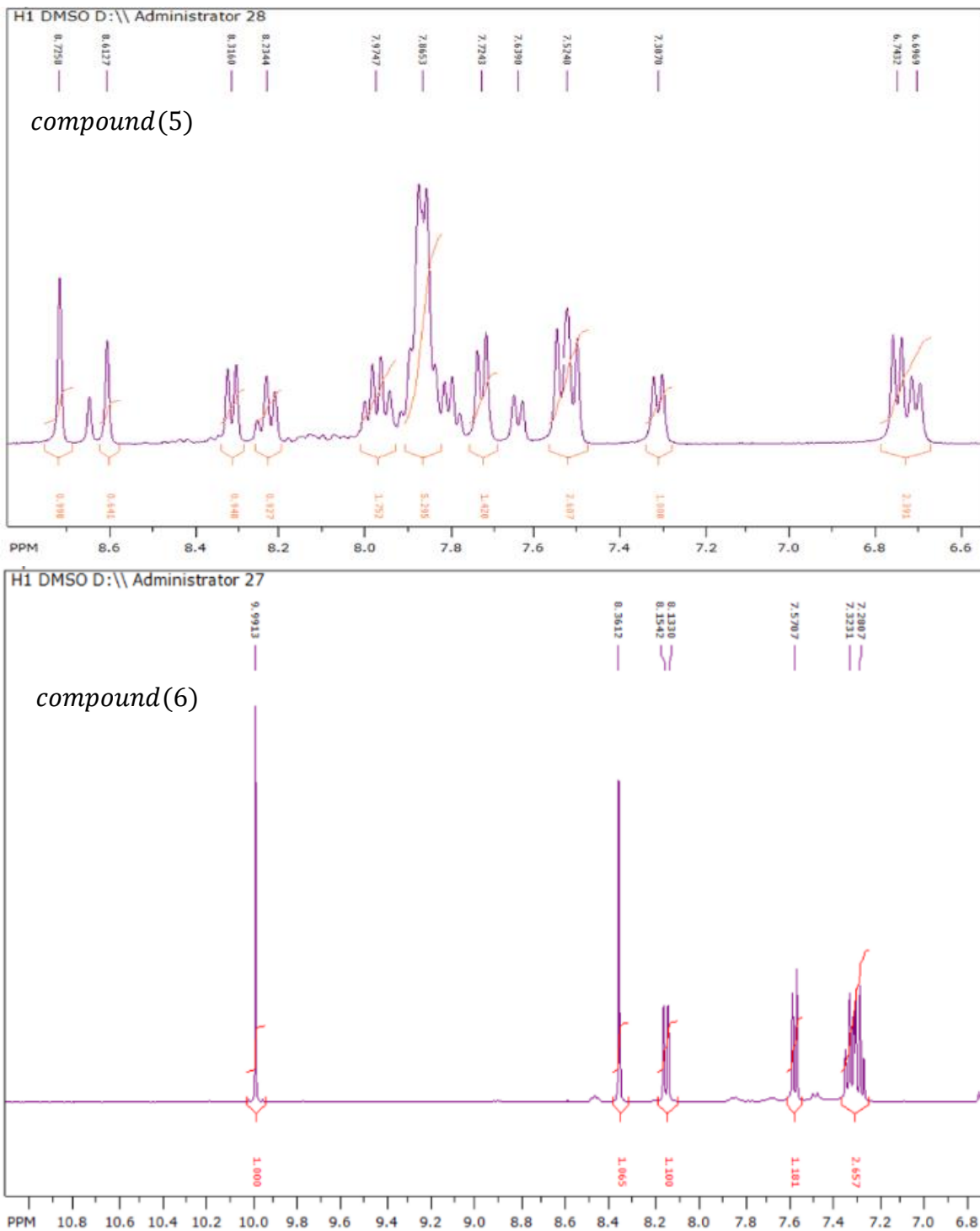
2. HNMR Spectra

The HNMR spectral data cited in experimental part. the formation of Schiff bases are confirmed by the appearance of a sharp signal at the region (8.83 - 9.1 ppm). Which attributed to azomethine proton (HC=N). The two hydroxyl group in compound (2) which formed from the 2-hydroxyl-3-methoxy benzaldehyde and benzidine Show only one singlet sharp signal at (13.42 ppm) which indicate that the two hydroxy group are equivalent. While the two hydroxy proton in compound (3) resulting from the

condensation of Salicylaldehyde with benzidine show also one broad signal at (13.235 ppm). This may be due the formation of hydrogen bonding between the OH group with the C=N group (Ebrahimi et al, 2013; Samal et al, 1999; Soltani et al, 2015). The H-NMR spectrum of compound (6) which resulting from the condensation of indole-3-carboxaldehyde and benzidine show singlet signal at 9.9ppm which attributed to equivalent two NH proton of indole moiety (Nag et al, 2001). The H-NMR spectra of the Schiff bases is shown in Figure (2).







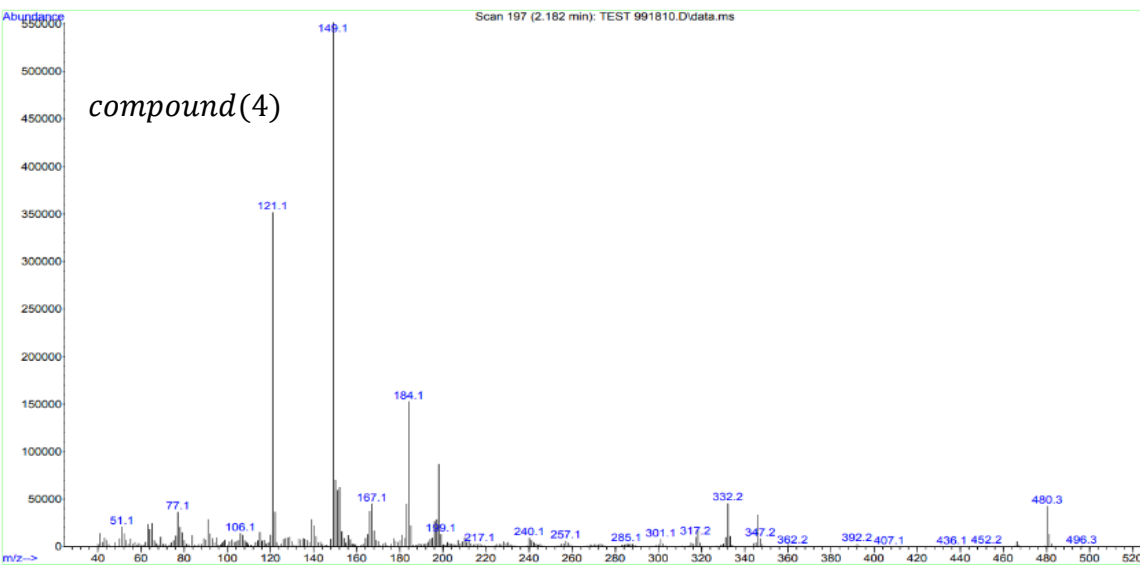
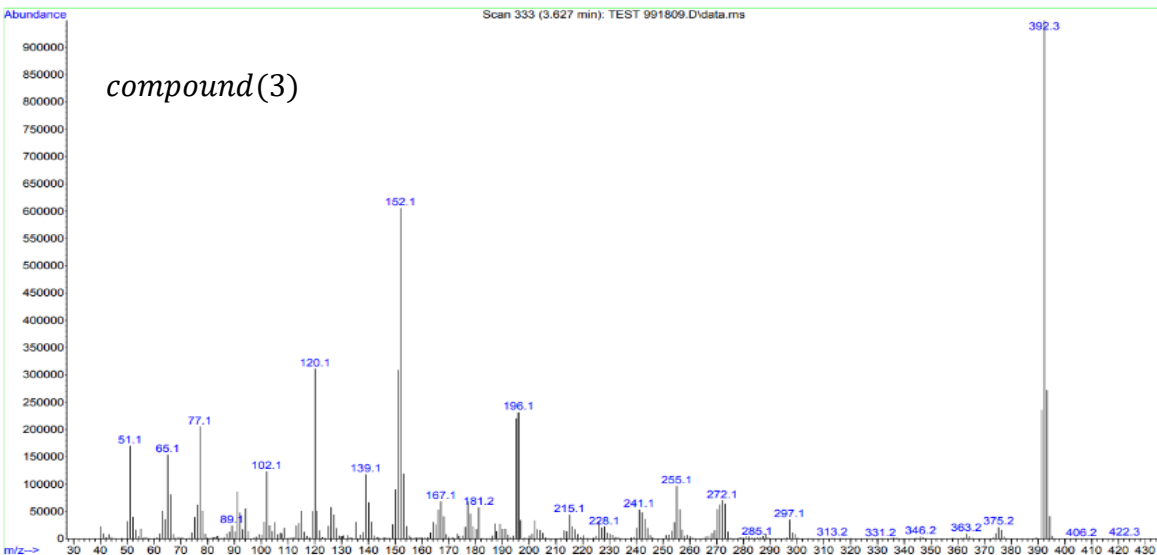
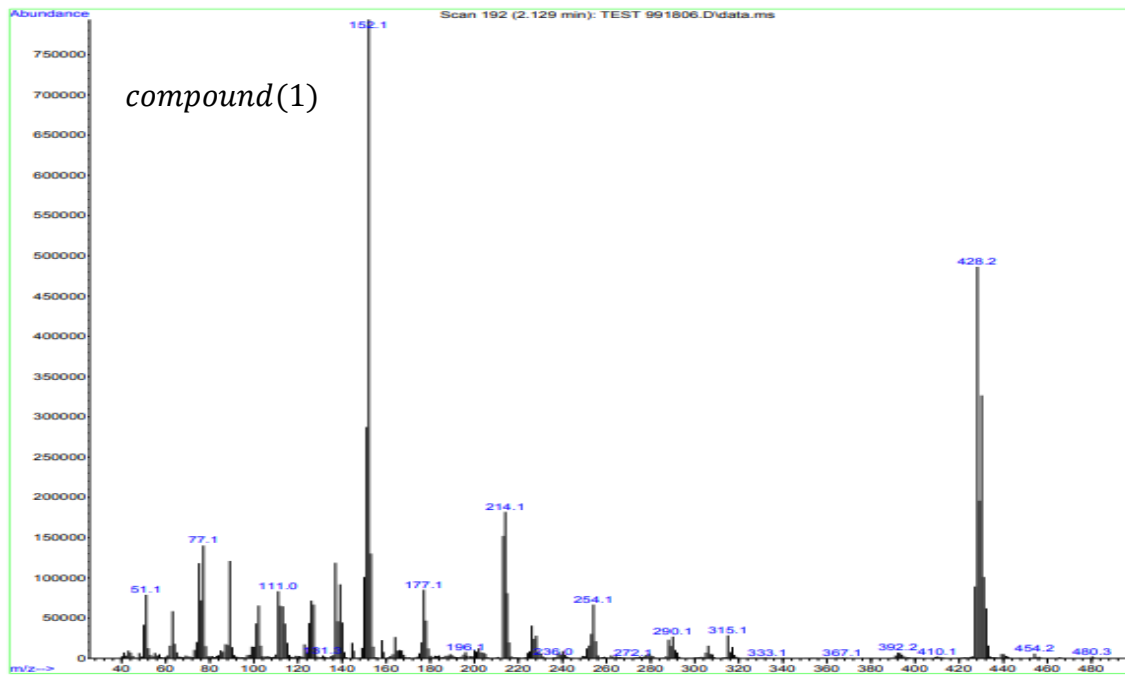
Figures 2. 1H-NMR- spectrum of compounds (2-6)

3. Mass Spectra

The mass spectra of all compounds shows a molecular ion peak at m/z confirmed (Figure 3). The condensation of 2 mole of aldehydes with 1 mole of benzidine, where compound (1) show the molecular ion at $m/z=428$ (R.I=62%). Compound (2) show a

molecular ion peak at $m/z=452$ (R.I 6%), compound (3) show a molecular ion peak at $m/z=392$ (R.I 100%) which represent the base peak and reflect the high stability of M^+ , compound (4), the show molecular ion peak is observed at $m/z= 480.3$ (R.I 9%) and compound (5) is observed at $m/z=560.3$ (R.I 6%).





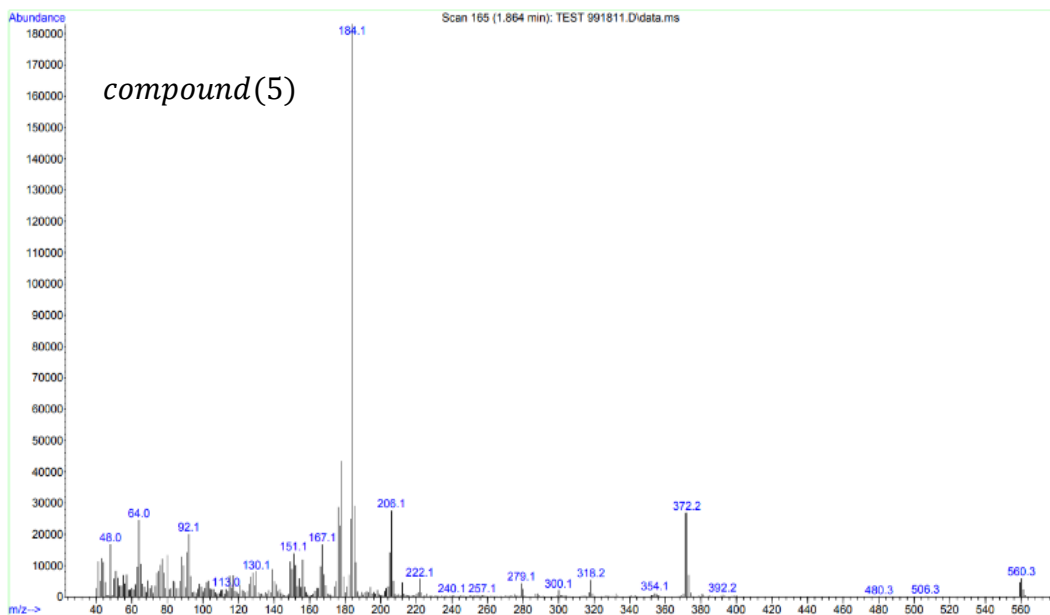


Figure 3. The mass spectra of synthesized compounds (1,3,4 and 5)

4. Florescence Spectra

The high aromaticity of prepared Schiff base and condensed fused ring, the florescence properties was suspected. So the florescence spectra of all prepared Schiff bases were studies at room temperature as DMF solution shown in Figure(4). (Yuste, 2005). The Schiff bases of benzidine with various aromatic aldehyde including (chloro, hydroxy and methoxy aldehyde), the Emission maximums shift compounds 4 and 5 from 420 nm and 410 nm in agreement with the decrease in the conjugation of the Ar-substituent from phenanthrene to para, ortho methoxy- substituted phenyl. While the presence of the electron withdrawing chloro group in compound (1) and the electron donor phenyls substituted with dimethoxy groups in compound (4), maybe a withdrawing character to the compound. While, the phenyl-dimethoxy in compound (4) resulted to exhibit a stronger donor effect, since there is a wavelength difference of 16 nm of among them (Kaya and Yilmaz, 2017), The Florescence spectra data of the Schiff bases is shown in Table 1.

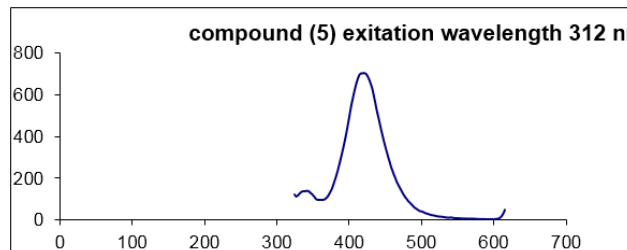
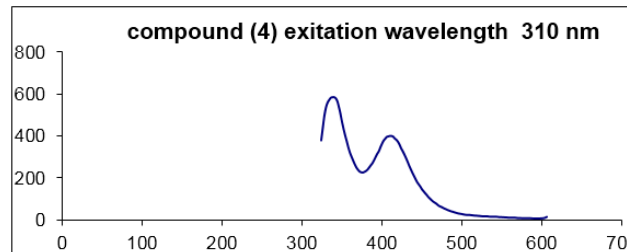
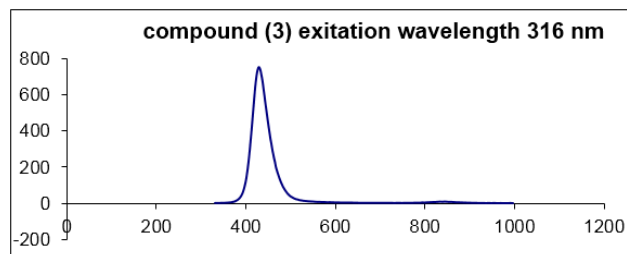
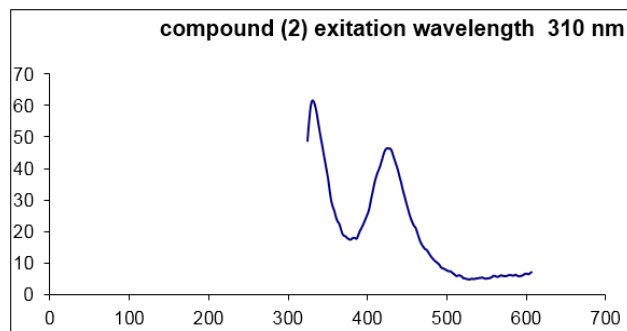
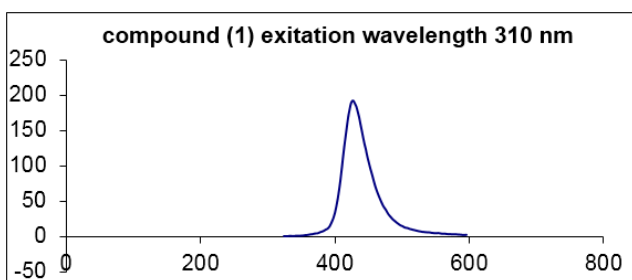


Figure 4. Emission spectra of compounds(1-5)



Table 1. Fluorescence spectra related data of compounds (1-5)

Sample Name	Excitation wavelength (nm)	Emission wavelength (nm)	Stoke's shift $\Delta \lambda_{ST}$
Compound(1)	310	426	116
Compound(2)	310	423	113
Compound(3)	316	428	112
Compound(4)	310	410	100
Compound(5)	312	420	108

5. Thermal Analysis Studies

The TG/DTA curves of prepared compounds shows in Figures (5). No mass lose up to were observed which indicated the high thermal stability. The thermal decomposition of all compounds occurs completely in one sharp step which was observed at 380°C for compound(1), 300°C for compound(2), 395°C for compound(3), 391°C for compound(4), 452°C for compound(5). (Hadi, 2015; Ahmad et al, 2020).

The leaving residual up to 600° C, Which represent carbon as final residue (Abd El-Wahed et al, 2009). The kinetic parameters were evaluated using Coat-Redfern methods of calculation in the following from.

$$\text{Log} \left[\frac{\log \frac{w_f}{w_f - w_t}}{T^2} \right] = \text{log} \left[\frac{AR}{\theta E} \left(1 - \frac{2RT}{E} \right) \right] - \frac{E}{2.303RT} \quad (1)$$

Where W_f and W_t are weight loss at the end of stage and weight loss at temperature (t), respectively. E, R, A and θ are the activation energy, the universal gas constant, pre - exponential factor and heating rate (10 °C/min), respectively.

By plotting the left -hand side of equation (1) vis 1000/T was drawn, the E values were calculated from slope and A values were determined from the intercept. The enthalpy (ΔH), entropy (ΔS) and Gibbs free energy (ΔG) values were calculated using the following equations:

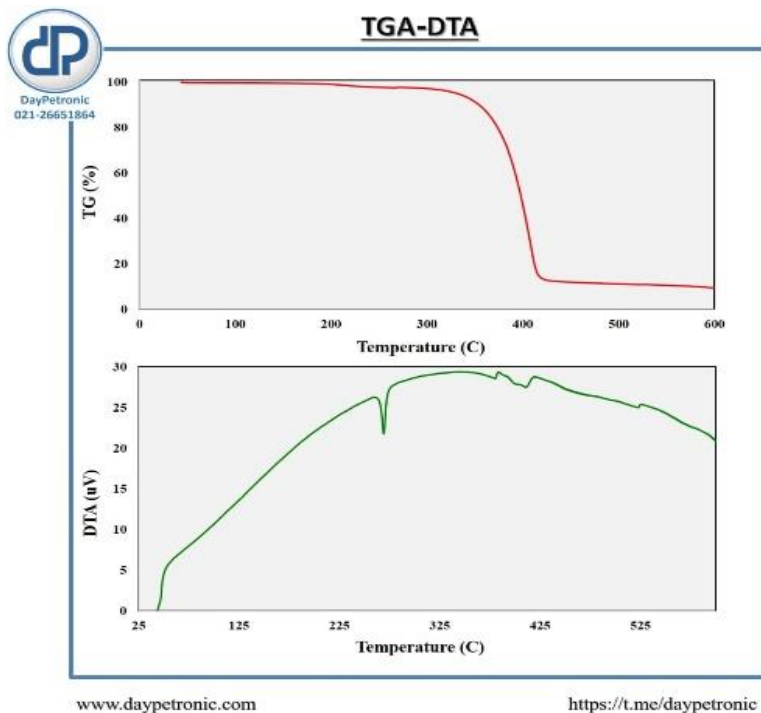
$$A = \left(\frac{K_b T}{h} \right) = e^{\Delta S/R} \quad (2)$$

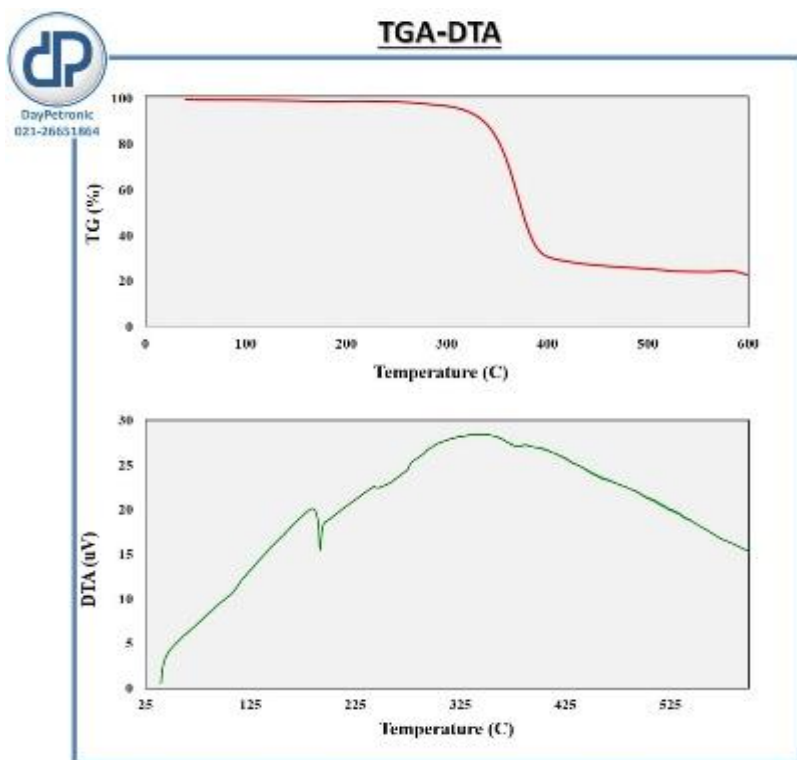
$$\Delta H = E - RT_m \quad (3)$$

$$\Delta G = \Delta H - T_m \Delta S \quad (4)$$

Where K_b is Boltzmann constant, h is plank constant and A is Arrhenius factor.

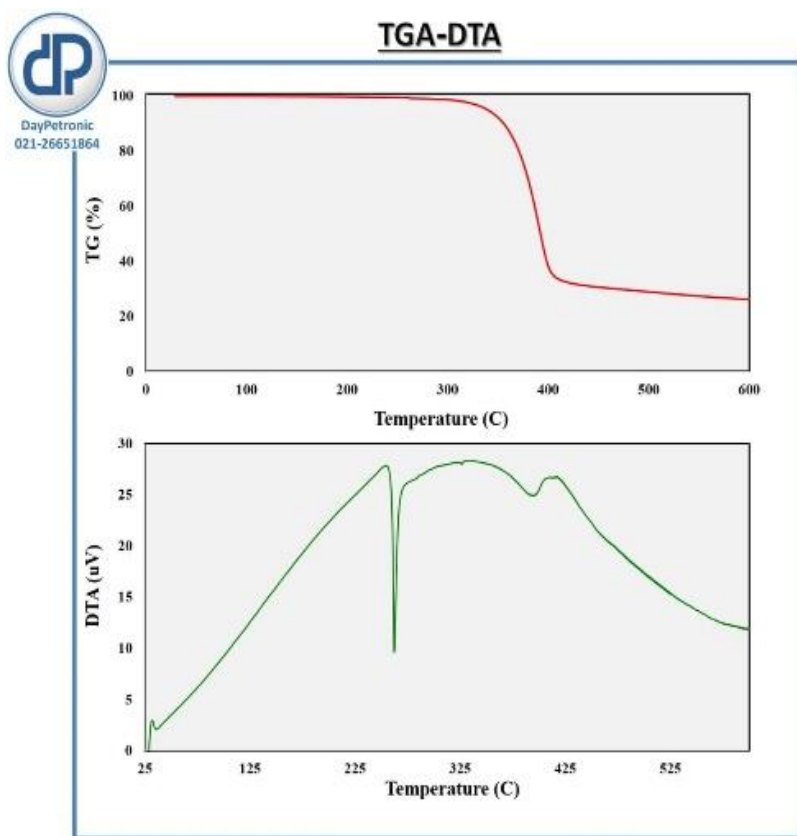
The values of correlation coefficients (r^2), Figure (6). Indicates the good agreement with experimental data where the values ranging from 0.9137–0.958. The activation energy (E) of decompositions were in the range 74.7464 to 109.0985 KJ mol⁻¹ and the high values indicate the compounds are thermally stable, the positive value of ΔG and negative value of ΔS reflect the nonspontaneous decompositions reaction (Coats and Redfern, 1964; Horwitz. and metzger, 1963; Ebrahimi et al, 2014). The kinetic parameters data are summarized in table (2).





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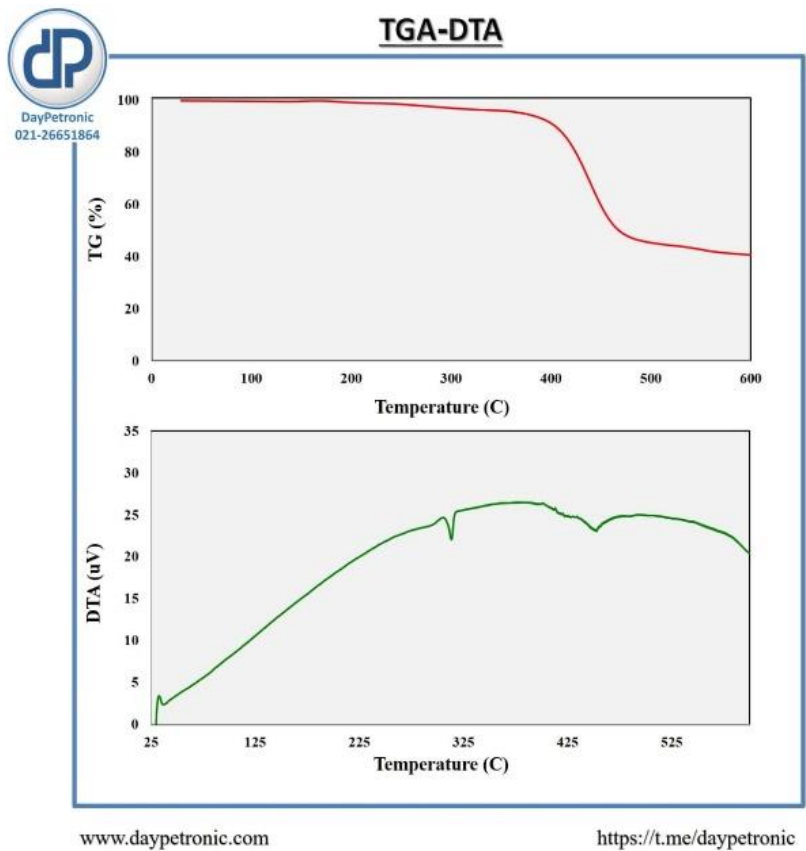
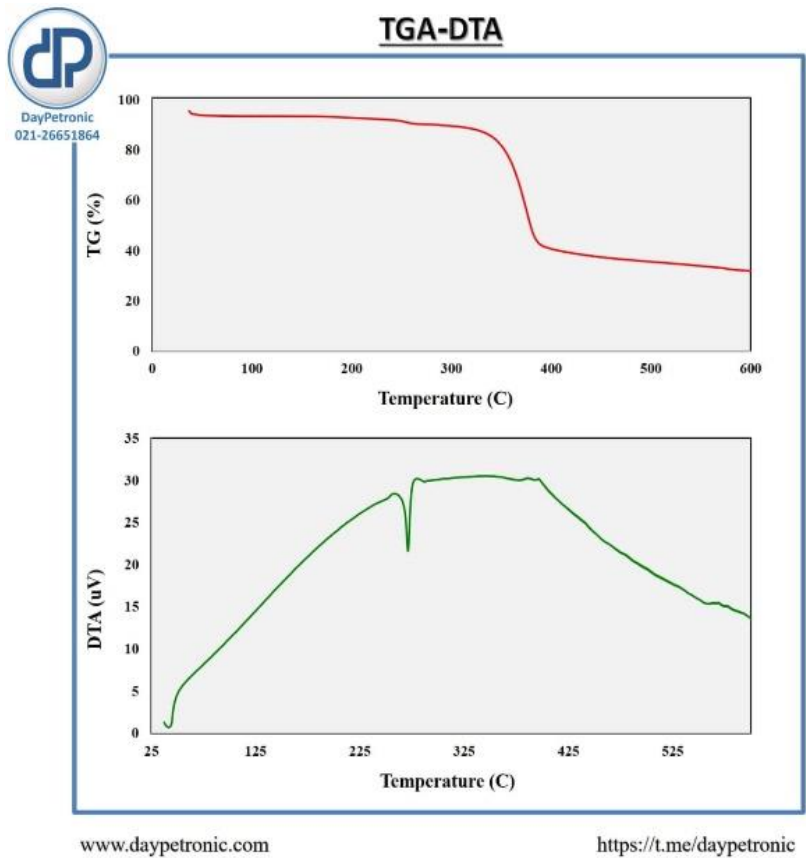
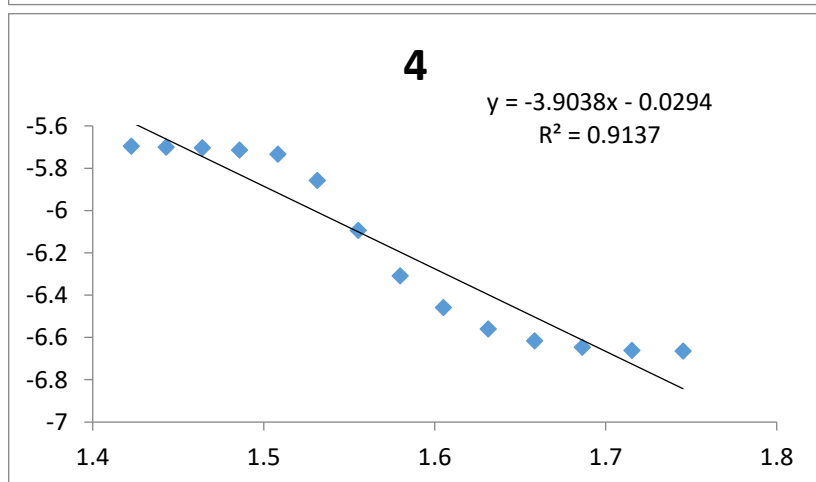
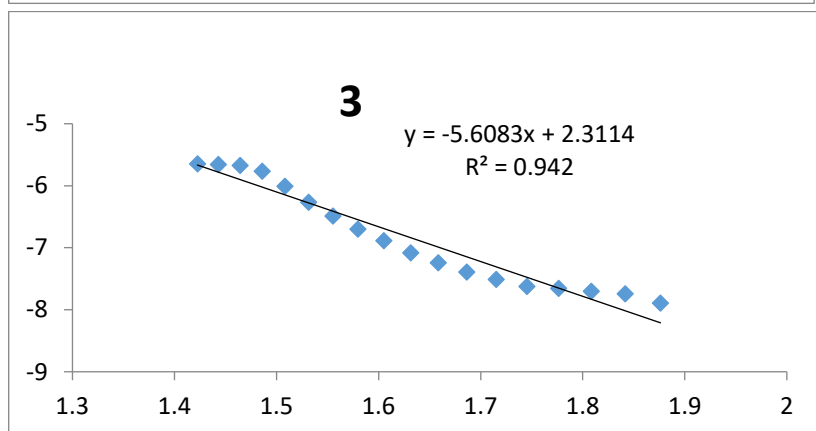
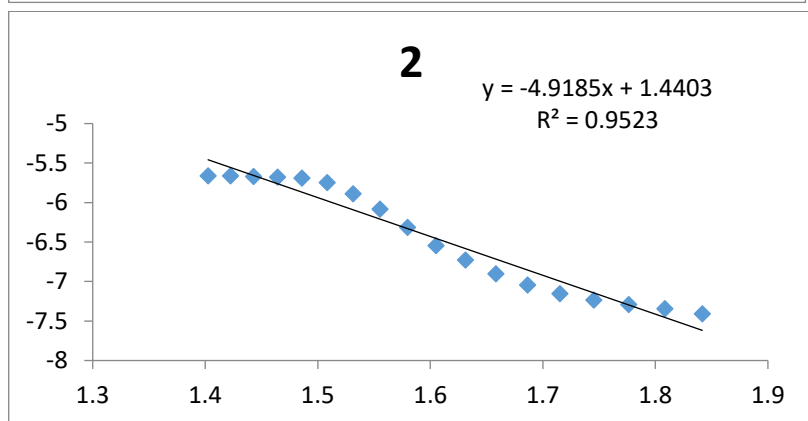
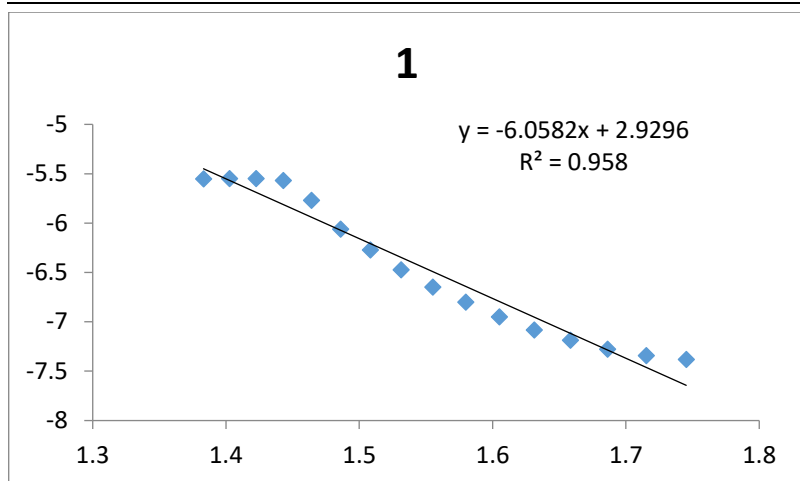


Figure 5. TG - DTA curve of synthesis compounds (1-5)





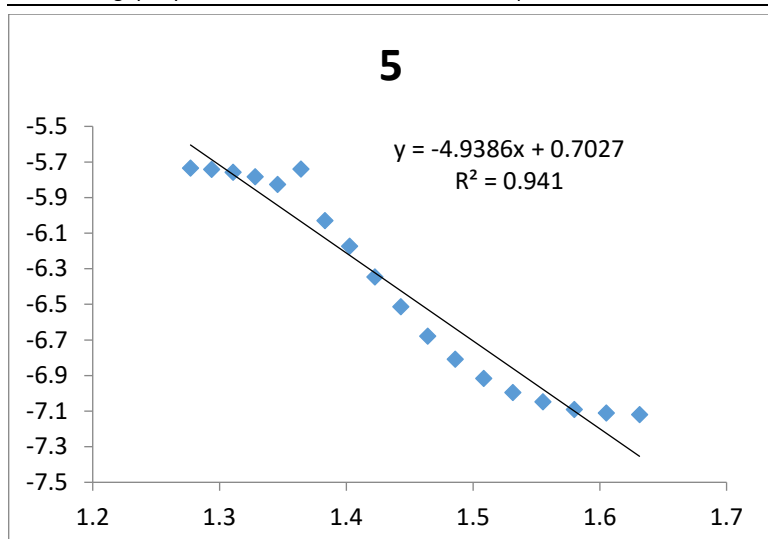


Figure 6. Kinetic curve of decomposition of compounds (1-5)

Table 2. Kinetic parameters of the thermal decomposition of compounds

Complex	A (S ⁻¹)	E (KJ/mol ⁻¹)	Δ H (KJ/mol)	ΔS (KJ/mol. K)	ΔG (KJ/mol)	r ²
1	537398.6	109.0985	104.5923	-0.14018	180.5698	0.958
2	52028.33	94.17522	90.31752	-0.1583	163.7691	0.952
3	440936	107.3833	102.9353	-0.14172	178.7537	0.942
4	1400.39	74.7464	70.22358	-0.18968	173.4088	0.913
5	9560.265	94.5595	89.67918	-0.17434	192.0174	0.941

Conclusions

Benzidine Schiff bases have been synthesized from different aldehyde and characterize by FT-IR, EI-Mass and H-NMR, TGA/DTA and UV-Vis and photoluminescence. The results support the suggested structure of benzidine Schiff bases. Thermal studies indicated high stability benzidine Schiff bases. Fluorescence studies showed that the reported compounds (1-5) can potentially serve as photoactive materials.

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