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Flammability of Polyester and Epoxy Resins by Using Some of a New Organic Compounds

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Abstract--- *In this work, seven additives were used, namely: (5-(6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxybenzaldehyde) (AdditiveI), (5-6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one) (AdditiveII), 5,6-(bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one (Additive III), 5-6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-methoxyphenyl)prop-2-en-1-one (Additive IV), 4-6-bromobenzothiazol-2-yl)diazenyl)-2-2-hydroxy-5-methylphenyl)imino)methyl) phenol(AdditiveV).4-6-bromobenzothiazol-2-yl)diazenyl)-2-2-hydroxy-5-methylphenyl)imino)methyl)phenol (Additive VI) and(1E)-N-(5-((6-bromobenzo[d]thiazol-2-yl)diazenyl)-2-hydroxybenzylidene)-N'-(4-chlorophenyl)formimidamide(Additive VII). The effects of these additives on flammability of some of the thermosetting polymers (unsaturated polyester and epoxy resins), have been studied. Sheets of the specimens (resins with different weight percentages of additives), in dimensions (150X150X3) mm were prepared. Two standard test methods used to measured the flame retardation which are: (ASTM: D-2863) and (ASTM: D-635). Results are obtained from these tests indicated that, additive 6A has high efficiency as flame retardant, self–extinguishing (S.E.) at the percentage (0.1 %) for unsaturated polyester and the epoxy resins. Also self-extinguishing (S.E.) at the percentage (0.1%) for additives 1A, 4A and 5A, in unsaturated polyester resin and non-burning occurs in 0.15% with Additive 6A and 5A in unsaturated polyester resin but it is occur in 0.2% with additives 6A and 5A in epoxy resin. Additive A shows low effect on flammability in both resins.*

Keywords--- *Flame Retardant, Flammability, Polyester Resin, Unsaturated Polyester Resin, Epoxy Resin, Schiff Base, Chalcones.*

I. INTRODUCTION

The history of all polymer materials was traced by the success of their applications in replacing tradition materials like wood, leather and metals[1]. The rapidly expansion of combustion in courage many researches to used many additives to retard flammability of the polymers [2].A large number of synthetic polymeric materials were used these days, with various different properties are available for medical applications and engineering matrices. Most of the common materials have sufficient mechanical stability and elasticity as well as desired stability towards degradation, and are non-toxic. [3, 4].Flame retardants are used to reduce flammability of polymeric materials, these chemical compounds are capable of imparting flame resistance to the materials and they can be classified into two general types [5] : those which do not react chemically with the polymer and the other type which are those incorporated chemically in to the basic polymer structure. A good flame – retardant additive must meet following

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requirements [6, 7]: thermally stable up to the processing temperature of the polymer and stable to light, not interact with main chain of the polymer, should not be poisonous and should not inversely affect physical properties of polymer. Many inorganic compounds[8, 9]were used as flame retardants, such as antimony, phosphorus and halogen compounds, but organic compounds were not used as flame retardants because they needed certain preparation conditions and their high efficacy was discouraged inhibition. In this work, the effectiveness of some organic aromaticshiff bases and Chalcones compounds was studded.

II. EXPERIMENTAL PART

1-Materials

A- All chemicals were used in this work analytical grade.

B- Preparation of flame-retardant additives

1- Preparation additive A

First step: Preparation compound (6-bromobenzothiazol-2-amine)[10]

A mixture (4.3 g, 0.25 mole) of para-bromoaniline and (3.80 g, 0.5 mole) of potassium thiocyanate in 70 ml of glacial acetic acid and then refrigerate the mixture to (0-5 C^o) and on the other hand, a mixture of 1.3 ml of bromine dissolved in 30 ml of glacial acetic acid was added with a continuous stirring until the addition was completed. Leave the mixture for an hour to settle at (0-5 C^o) and then gradually cool the distilled water. A cold, concentrated solution of sodium hydroxide was gradually added to the formed solution and a yellow light-colored trace was observed, filtered and washed with water several times and then re-crystallization with ethanol. The reaction process was followed using TLC technology.

Second step: Preparation of additive (A)[11]

[5-(6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxybenzaldehyde]

Solution (I)... Preparation of the azocompound (A) from dissolving the Thiazole derivative which contain bromine atom by taking (1.44 g, 0.004 mole) and dissolving it in 5 ml of concentrated hydrochloric acid and 20 ml of distilled water in a round bottom flask and then return the mixture in a snow bath at (0-5 C^o).

Solution (II)... dissolved (0.6 g) of sodium nitrite in (5 ml) of distilled water and stirred the mixture for 20 min. at (0-5 C^o).

Solution (III)... This solution was prepared by added solution (II) to the solution (I) in form of droplets gradually added to obtain the diazonium salt at (0-5 C^o).

Solution (IV)... Added 0.6 ml of salicyldehyde to a solution which component (1.0 g of sodium hydroxide dissolved in 50 ml of distilled water and 5 ml of ethanol) and cool the mixture at (0-5 C^o).The solution (IV) was then added to the solution (III) and the reaction was left for one hour at pH 6-7 and at (0-5 C^o), the product was filtered, the precipitate was collected and dried at 35 C° in the vacuum oven and thenre-crystallizationby using ethanol.

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2- Preparation of additive 1A[12, 13]

5-6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one

This derivative was prepared by condensation (2.17 g, 0.006 mole) of azo compound (A) with(0.66 g, 0.004 mole) of(4-hydroxy-3-methoxy acetophenone) in a 100 ml of round bottom flask by dissolved it in 50 ml ethanol and stirred for 20 min.; A 10% sodium hydroxide solution was gradually added to the droplet for 2-3 min. under continuous stirring.The mixture was refluxed at 35 C° for 8-16 hr. and pH was equal to 7 by adding 10 ml of concentrated hydrochloric acid (10% M). The product was filtered and washed with distilled water and re-crystallization with ethanol. The reaction process was followed using TLC technology.

3- Preparation of additive 2A[12, 13]

5,6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one

This derivative was prepared by condensation (2.17 g, 0.006 mole) of azo compound (A) with(0.54 g 0.004mole) of(4-hydroxy acetophenone) in a 100 ml of round bottom flask by dissolved it in 50 ml ethanol and stirred for 20 min.; A 10% sodium hydroxide solution was gradually added to the droplet for 2-3 min. under continuous stirring.The mixture was refluxed at 35 C° for 8-16 hr. and pH was equal to 7 by adding 10 ml of concentrated hydrochloric acid (10% M). The product was filtered and washed with distilled water and re-crystallization with ethanol. The reaction process was followed using TLC technology.

4- Preparation of additive 3A[12, 13]

5-6-bromobenzothiazol-2-yl)diazenyl)-2-hydroxyphenyl)-1-(4-methoxyphenyl) prop-2-en-1-one

This derivative was prepared by condensation (2.17 g, 0.006 mole) of azo compound (A) with(0.6 g 0.004mole) of(4-methoxyacetophenon) in a 100 ml of round bottom flask by dissolved it in 50 ml ethanol and stirred for 20 min.; A 10% sodium hydroxide solution was gradually added to the droplet for 2-3 min. under continuous stirring.The mixture was refluxed at 35 C° for 8-16 hr. and pH=7 by adding 10 ml of concentrated hydrochloric acid (10% M). The product was filtered and washed with distilled water and re-crystallization with ethanol. The reaction process was followed using TLC technology.

5- Preparation of additive 4A[14, 15]

4-6-bromobenzothiazol-2-yl)diazenyl)-2-2-hydroxy-5-methylphenyl)imino) methyl)phenol

The derivative (4A) was prepared by condensation of azo compound (A) by dissolving (3.52 g, 0.009 mole) from (A) in 20 ml of absolute ethanol in a round flask (100 ml) and stirred for 10 min.. A solution of (0.49 g, 0.004 mole) from 2- amino-5- methyl phenol which dissolved in 15 ml of ethanol was added and mixed until the mixture is homogenized, then added 3 drops from glacial acetic acid to solution that formed which be dark color after several minutes from stirring and leaving the reaction to 3 hr. with continues stirring. The reaction process was followed using TLC technology and kept the product 24 hr. then re-crystallize by using ethanol.

6- Preparation of additive 5A[14, 15]

4-6-bromobenzothiazol-2-yl)diazenyl)-2-2-hydroxy-5-methylphenyl)imino) methyl)phenol

The derivative (5 A) was prepared by condensation of azo compound (A) by dissolving (3.52 g, 0.009 mole) from (A) in 20 ml of absolute ethanol in a round flask (100 ml) and stirred for 10 min. A solution of (0.69 g, 0.004 mole) from -amino-6- methyl pyridine which dissolved in 15 ml of ethanol was added and mixed until the mixture is homogenized, then added 3 drops from glacial acetic acid to solution that formed which be dark color after several minutes from stirring and leaving the reaction to 3 hr. with continues stirring. The reaction process was followed using TLC technology and kept the product 24 hr. then re-crystallize by using ethanol.

7- Preparation of additive 6A[14, 15]

(1E)-N-(5-((6-bromobenzo[d]thiazol-2-yl)diazenyl)-2-hydroxybenzylidene)-N'-(4-chlorophenyl)formimidamide

The derivative (6A) prepared by condensation of azo compound (A) by dissolving (3.52 g, 0.009 mole) from (A) in 20 ml of absolute ethanol in a round flask (100 ml) and stirred for 10 min..

A solution of (0.51g, 0.004 mole) from 4-chloro aniline which dissolved in 15 ml of ethanol was added and mixed until the mixture is homogenized, then added 3 drops from glacial acetic acid to solution that formed which be dark color after several minutes from stirring and leaving the reaction to 3 hr. with continues stirring. The reaction process was followed using TLC technology and kept the product 24 hr. then re-crystallize by using ethanol.

C. Polymers

1- Epoxy resin, type (CY223), hardener type (HY 956), imported from Ciba-Geigy Co.

2- Unsaturated polyester resin, hardener type (MEKP), imported from United Arab Emirate (U.A.E).

2-Standard Tests

A- ASTM: D-2863: The measurement of limiting Oxygen Index (LOI), is widely used for measuring flammability of polymers [16].

B- ASTM: D-635 : The measurement of rate of burning (R.B), average extent of burning (A.E.B), average time of burning (A.T.B), Self - Extinguishing (S.E) and Non – burning (N.B.) [17].

3- Preparation of polymeric specimens

The specimens of polymeric material containing additives were prepared in dimensions (150X150X3)mm; three sheets were prepared from each percentage weight (0.1, 0.15, 0.2, 0.25& 0.3 %); of flame retardant materials (as additives) and using the hardener for each resin. These sheets were cut as samples according to ASTM standard were used in this work.

III. RESULTS AND DISCUSSION

1- Preparation additive A

First step: Preparation compound (6-bromobenzothiazol-2-amine), **Figure (1),** FT-IR spectrum [18]showed**,** The peak appearance at (3271‹448) cm^{-1} returns to the NH$_2$ group, and the appearance of the peak at the frequencies (3093) cm $^{-1}$ to alkyl aromatic C-H stretching, while the peak at (1303) cm^{-1} is returned to the aldehyde C-N. The frequency band at (810) cm^{-1} indicates the presence of bromine in the Thiazole ring and, the appearance of the peak at the frequencies (694) cm^{-1} to C-S bond stretching, also the frequency band at (1635) cm^{-1} for C = N. We also note that there are two peaks that return to the presence of C=C group at frequencies (1527, 1442) cm^{-1}. Equation (1), represent this reaction.

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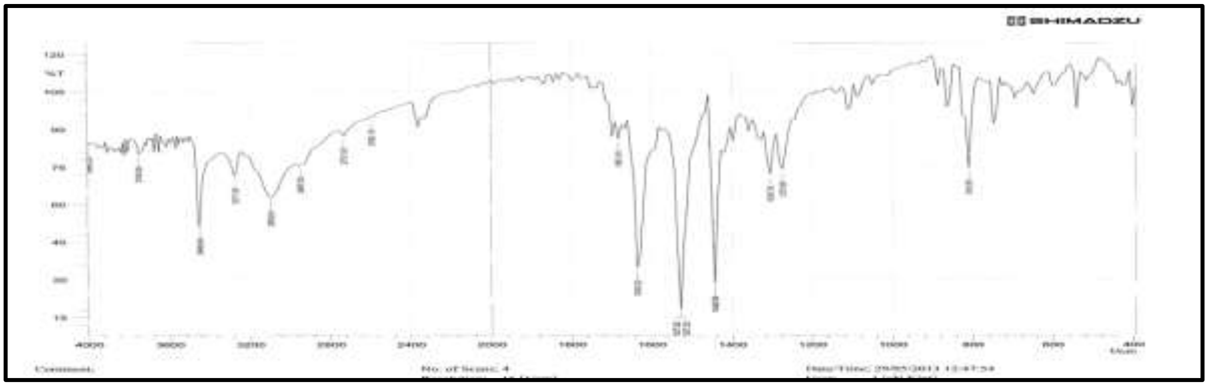
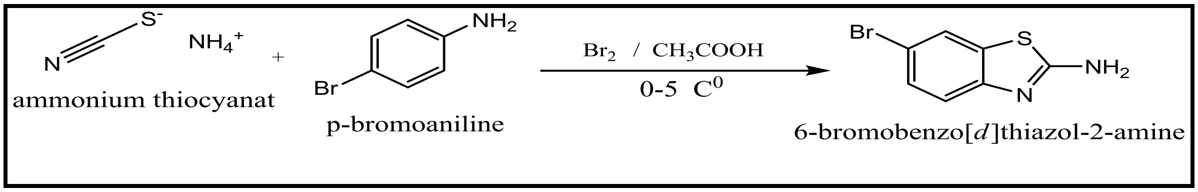


Figure 1: The FT-IR spectrum of compound 6-bromobenzothiazol-2-amine



Equation 1: Synthesis of 6-bromobenzothiazol-2-amine

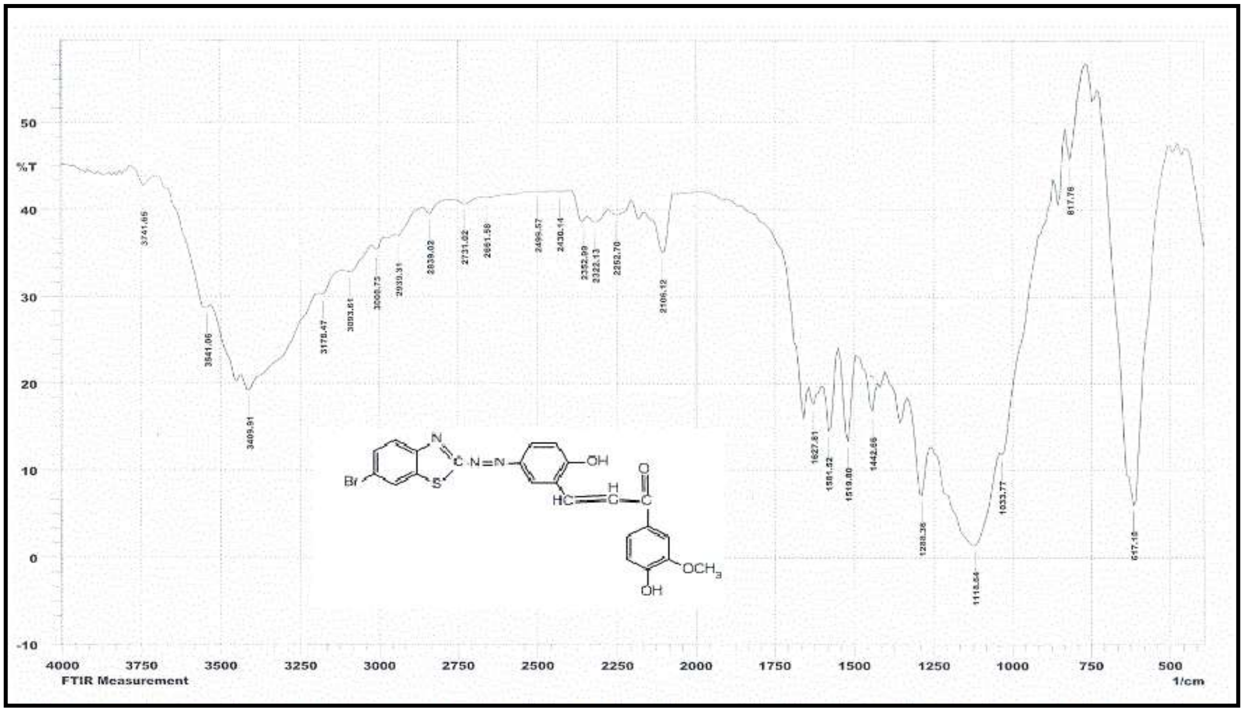


Figure 2: The FT-IR spectrum of compound A

Second step: Preparation of additive (A), **Figure (2),** The FT-IR spectrum showed; The peak appearance at (3309) cm $^{-1}$ returns to the OH phenol group, and the appearance of the peak at the frequencies (3086) cm^{-1} to alkyl aromatic C-H stretching, while the peak at (1843) cm^{-1} is returned to the aldehyde C=O. The frequency band at (678) cm^{-1} indicates the presence of bromine in the Thiazole ring and the frequency band at (1670) cm^{-1} for C=N. We also note that there are two peaks that return to the presence of the azo group at frequencies (1519, 1573) cm $^{-1}$.

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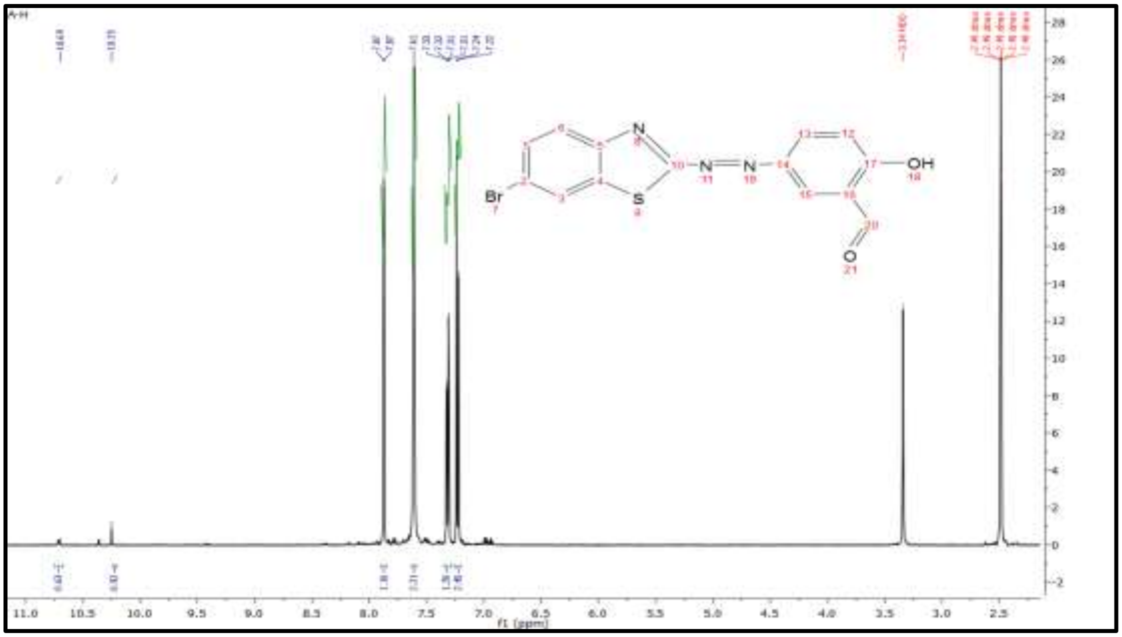

Figure 3: The ^{1}H-NMRspectrum of compound A

Figure(3), The spectrum of ^{1}H-NMR[19], showed; A single beam at the displacement of (10.69) ppm is attributable to the proton phenolic hydroxyl group associated with the ring, a double signal beam at displacement of (10.25)ppm is due to the aldehyde group (CHO) and another multiple signal at (7.2-7.8) ppm, return to the proton in the aromatic group (CH).

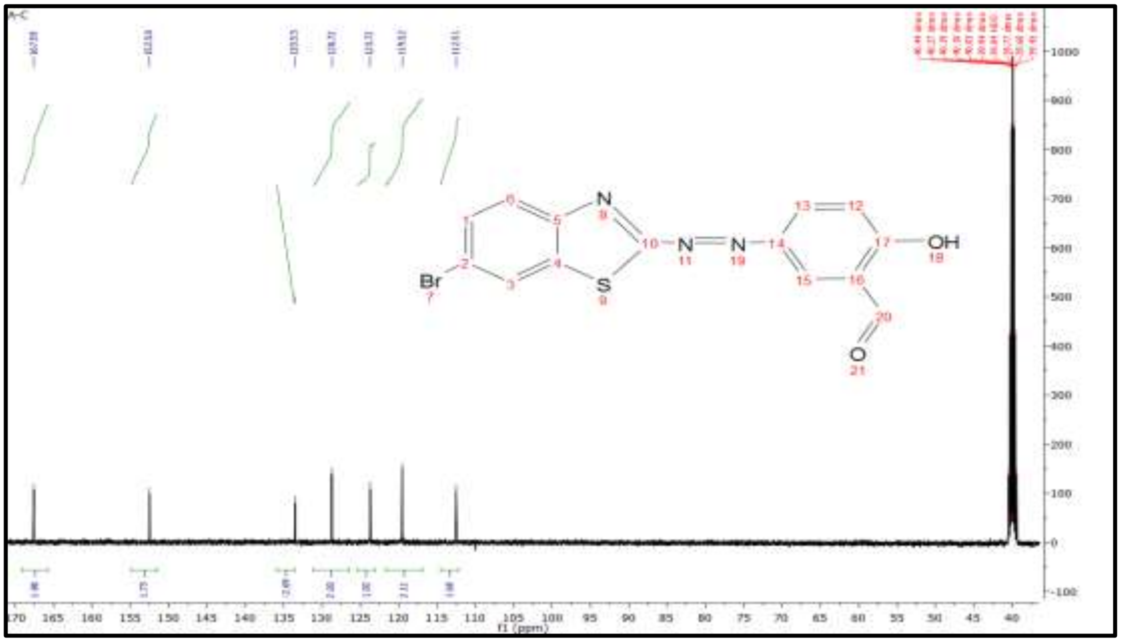

Figure 4: The ^{13}C-NMRspectrum of compound A

Figure(4),The spectrum of ^{13}C-NMR[20]showed, The signal at (133) ppm returns to C5, the signal at (127)ppm returns to C4and the signal at (190)ppm returns to C19, also the signal at displacement (152) ppm returns to C17, Signals at displacement at (112-123) ppm returns to carbon aromatic. **Equation (2),** represent this reaction.

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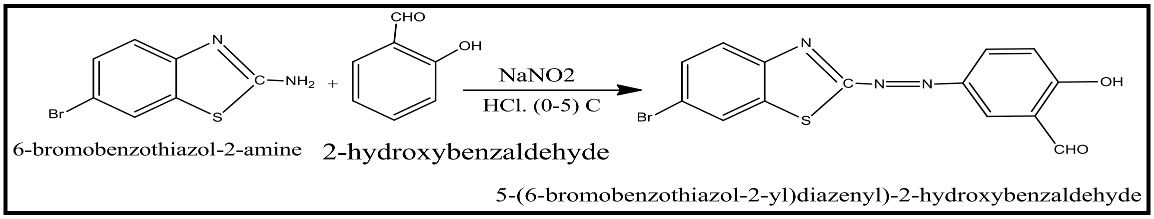

Equation (2): Synthesis of compound A

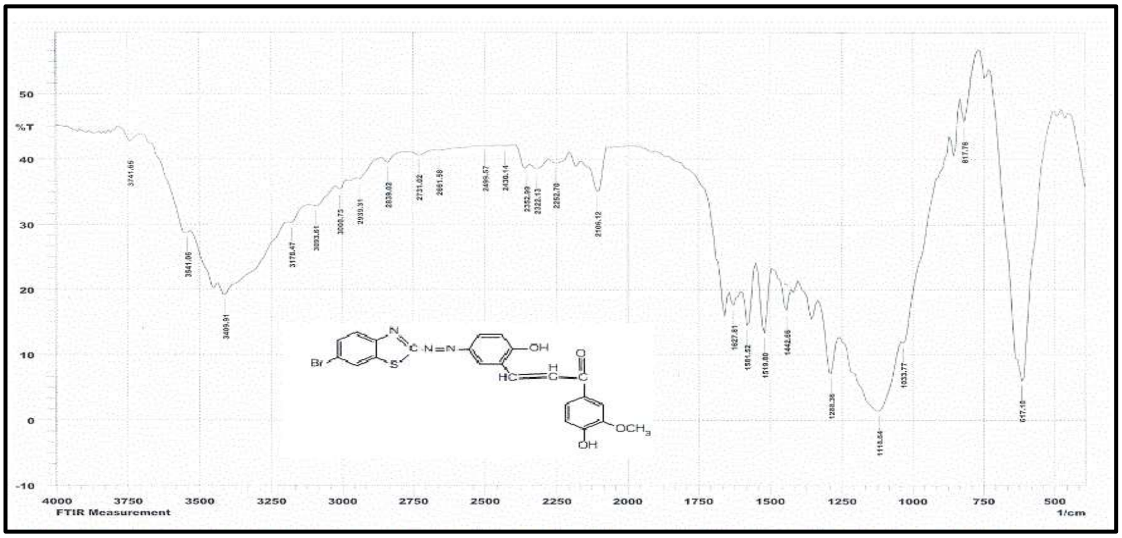

Figure (5): The FT-IR spectrum of compound 1A

2- Preparation of additive 1A; Figure(5), The spectrum of the FT-IR of the derivative (1A) showed; The peak appearance at (3309) cm $^{-1}$ to the stretching (OH), the peak at (3003) cm $^{-1}$ returns to the stretching (=CH)aromatic in the ring, the peak at frequency (1782) cm $^{-1}$ is due to (C=O) ketone, and the band at (570) cm $^{-1}$ indicates the presence of bromine in the Thiazole ring and the frequency band at (1658) cm $^{-1}$of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1496, 1512) cm $^{-1}$.

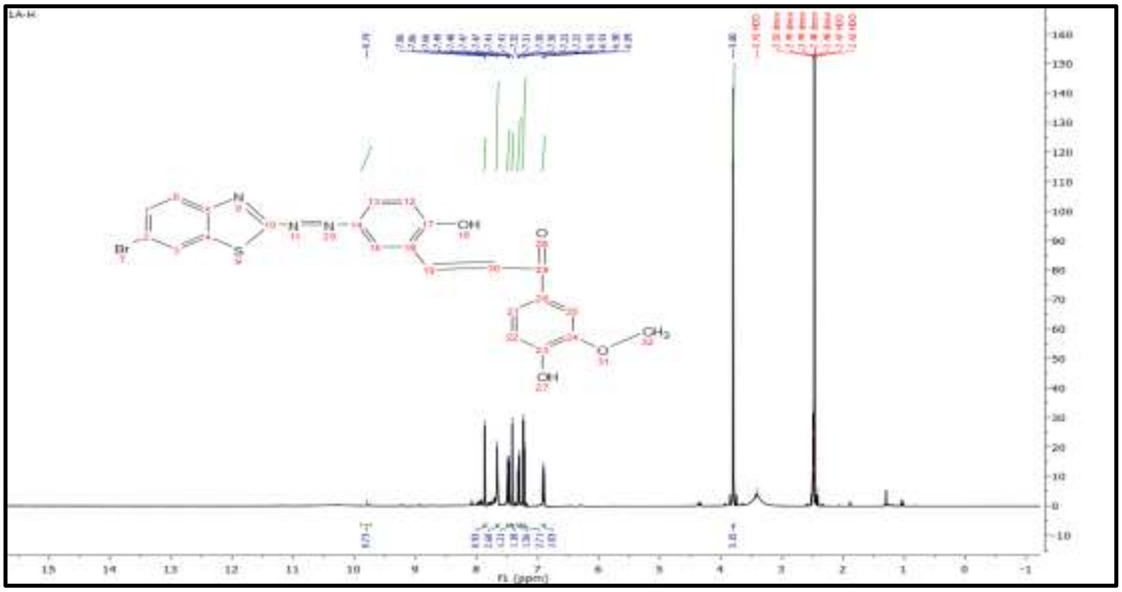

Figure 6: The ^{1}H-NMRspectrum of compound 1A

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Figure(6),The spectrum of 1H-NMR showed; Single signal at (9.75) ppm is return to the proton of phenolic hydroxyl group of the ring, the other signal at displacement (3.7) ppm return to the proton in the (OCH3) group, and two singles at the displacement (6.9,6.8) ppm of the alkenes group (CH=CH), and another multiple signal at (7.2-7.8) ppm of the protons aromatic group.

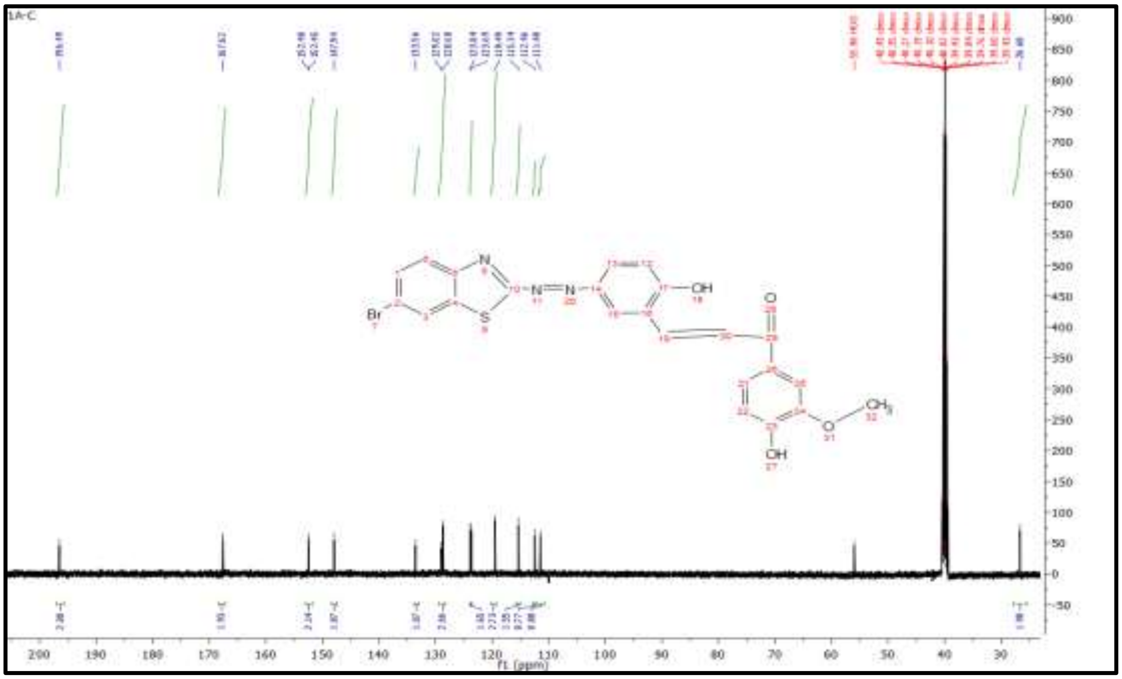
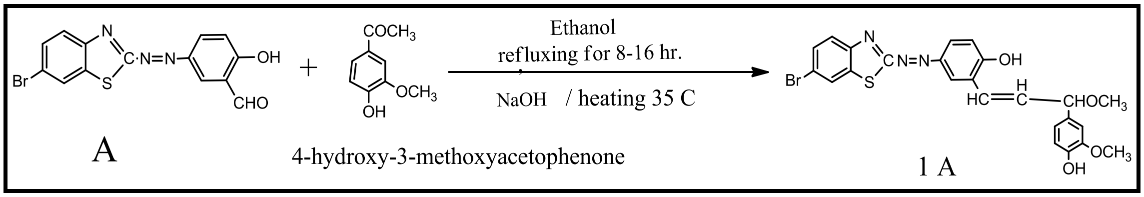


Figure 7: The ^{13}C-NMRspectrum of compound 1A

Figure(7),The spectrum of ^{13}C-NMR was also showed; Signals at (147.9) ppm return to C5, signal at (133.7) ppm return to C4and signal at (152.4) ppm return to C17, signal also at displacement (196.4) ppm return to C23, signals at displacement at (111-129) ppm returns to aromatic carbon**. Equation (3),** represent this reaction.



Equation 3: Synthesis of compound 1A (Chalcone)

3- Preparation of additive 2A; Figure (8), The spectrum of the FT-IR of the derivative (2A) showed; The peak appearance at (3417) cm^{-1} to the stretching (OH), the peak at (3249) cm^{-1} returns to the stretching(=CH)aromatic in the ring, the peak at frequency (1643)) cm^{-1} is due to (C=O) ketone, and the band at (617) cm^{-1} indicates the presence of bromine in the Thiazole ring and the frequency band at (1589) cm^{-1}of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1444, 1527) cm^{-1}.**Figure (9),**The spectrum of 1H-NMR showed; Single signal at (9.29) ppm is return to the proton of phenolic hydroxyl group of the ring, the other single at the displacement (4.5) ppm of the alkenes group (CH=CH), and another multiple signal at (7.2-7.8) ppm of the protons aromatic group. **Figure (10),** The spectrum of 13C-NMR was also showed; Signals at (129.9) ppm return to C5, signal at (129.9) ppm return to C4and signal at (168.8) ppm return to C17, signal also at displacement (131.07)

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ppm return to C29,signal at (162.6) ppm return to C13, signal at (114.7) ppm return to C30 signals at displacement at (114.9-129) ppm returns to aromatic carbon. **Equation (4),** represent this reaction.

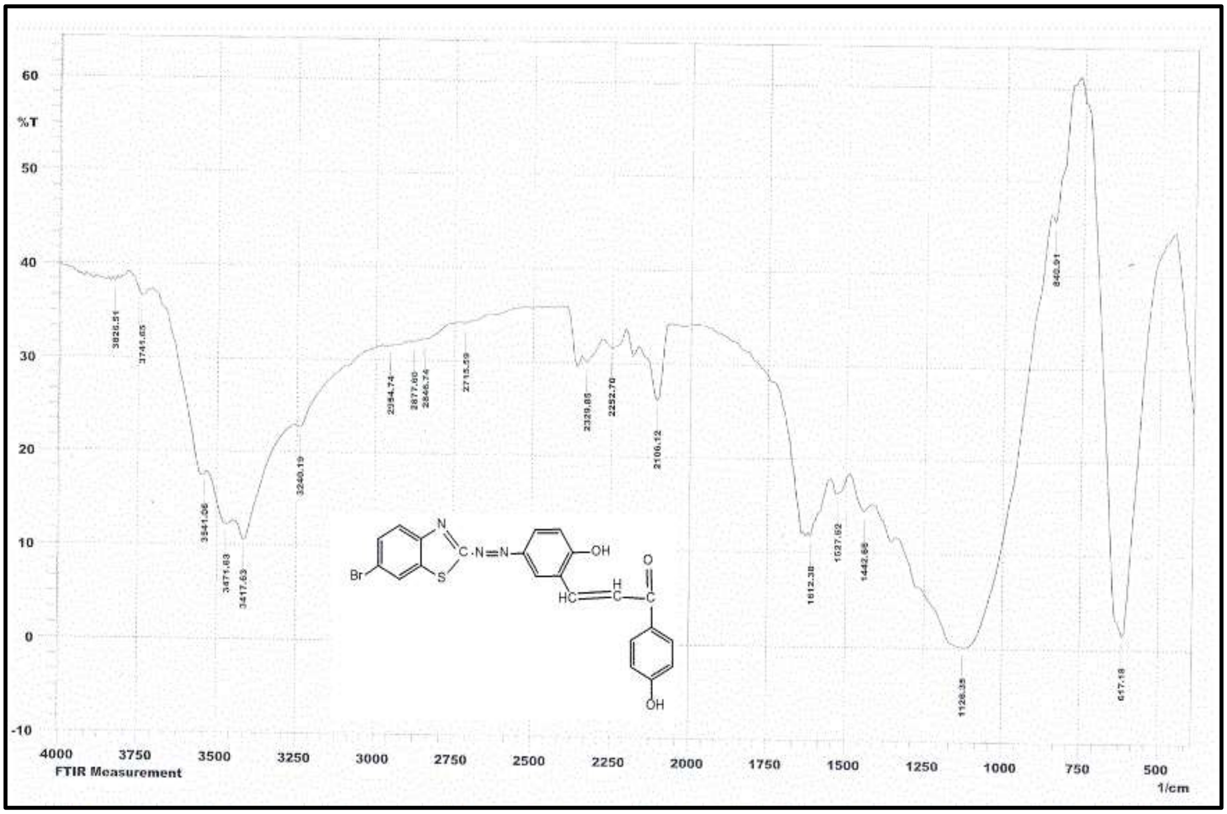

Figure 8: The FT-IR spectrum of compound 2A

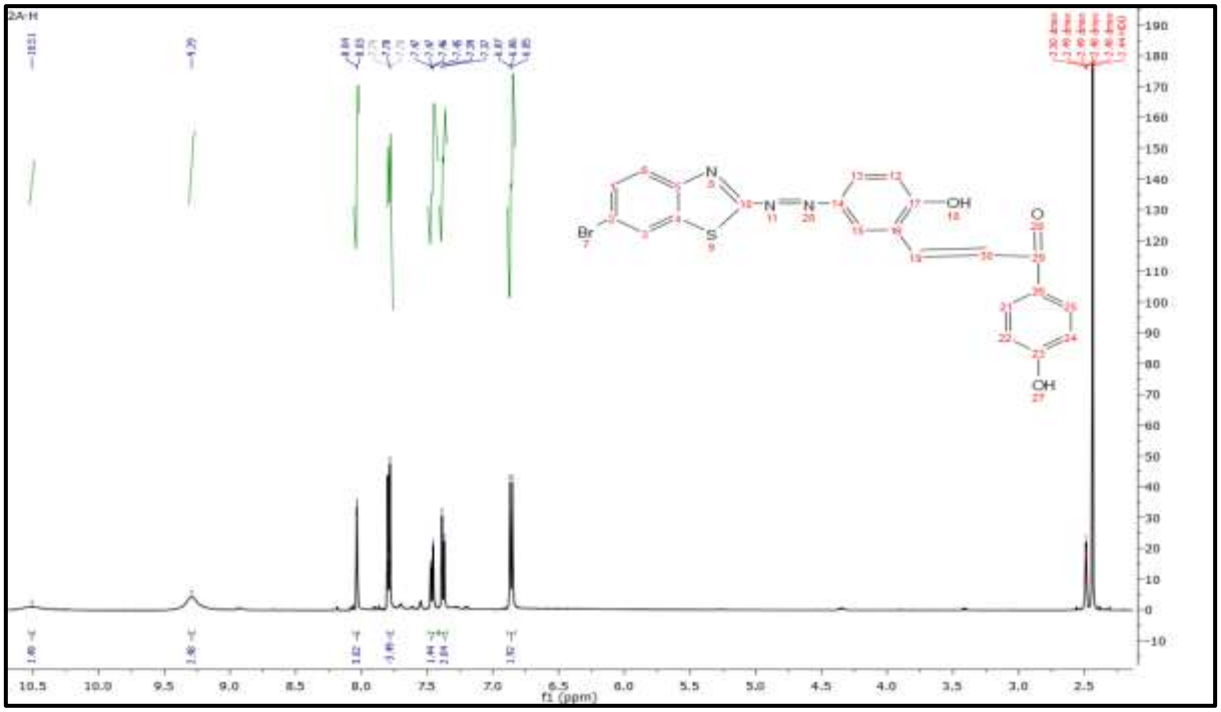

Figure 9: The ^{1}H-NMRspectrum of compound 2A

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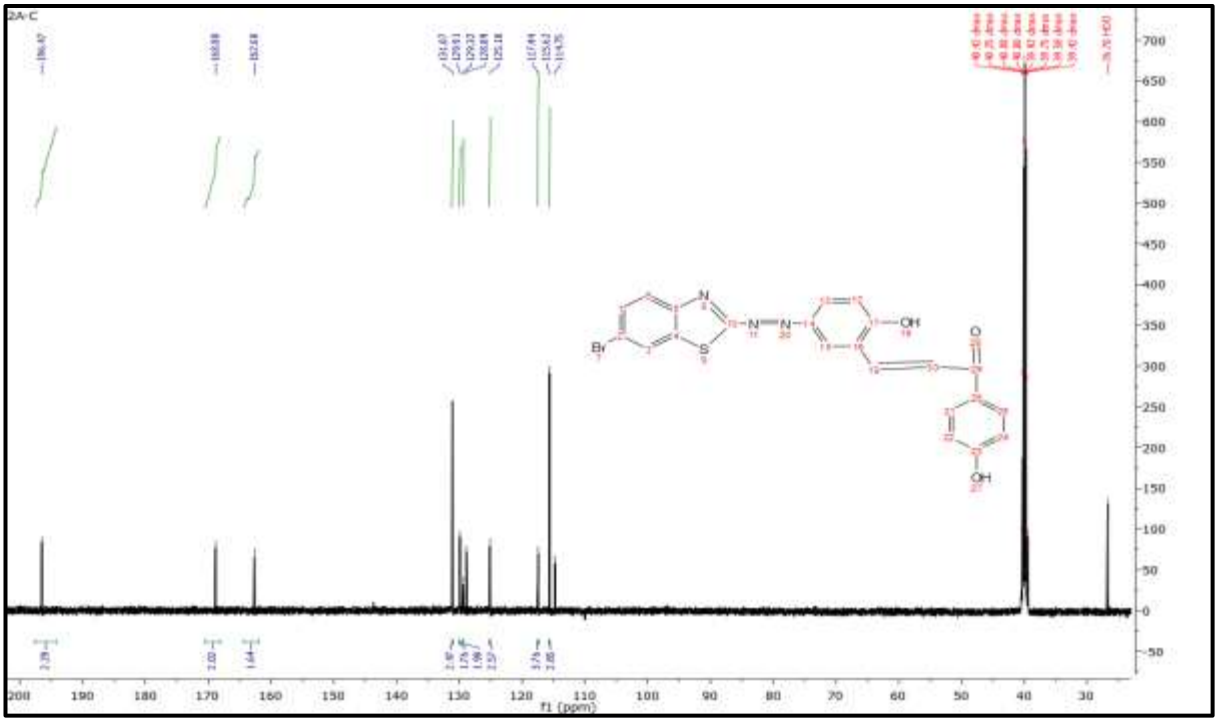
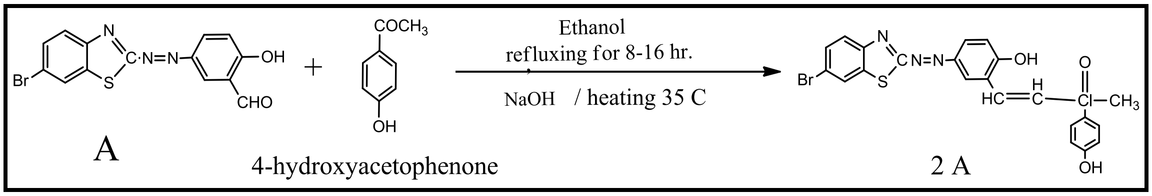
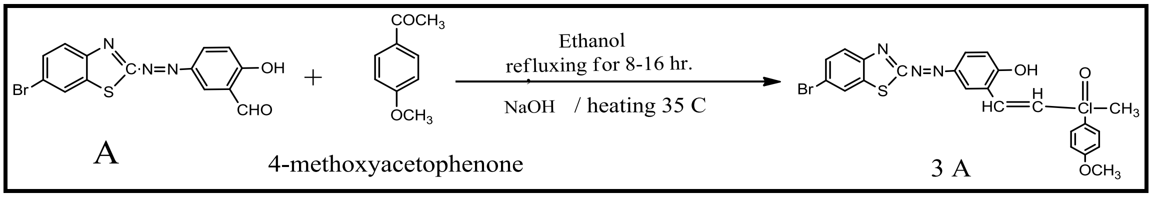


Figure 10: The ^{13}C-NMRspectrum of compound 2A



Equation 4: Synthesis of compound2A (Chalcone)



Equation 5: Synthesis of compound 3A (Chalcone)

3- Preparation of additive 3A; **Figure (11),**The spectrum of the FT-IR of the derivative (3 A) showed; The peak appearance at (3084) cm^{-1} to the stretching (OH), the peak at (3100) cm^{-1} returns to the stretching(=CH)aromatic in the ring, the peak at frequency (1658) cm^{-1} is due to (C=O) ketone, and the band at (677) cm^{-1} indicates the presence of bromine in the Thiazole ring and the frequency band at (1633) cm^{-1}of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1444, 1521) cm^{-1}.**Figure (12),**The spectrum of 1H-NMR showed; Single signal at (10.57) ppm is return to the proton of phenolic hydroxyl group of the ring, the other single at the displacement (3.8) ppm of the alkenes group (OCH3), and two singlets at displacement (6.9, 7) ppm return to (CH=CH), and another multiple signal at (7.8-8.6) ppm of the protons aromatic group. **Figure (13),**The spectrum of^{13}C-NMR was also showed; Signals at (141.09) ppm return to C_5, signal at (132.04) ppm return to $C_4$and signal at (159.03) ppm return to C_{17}, signal also at displacement (55.9) ppm return to C29,signal at (114.2) ppm return to

C_{30},signal at (172.2) ppm return to C_{14}signals at displacement at (117.2-130.8) ppm returns to aromatic carbon. Equation (5), represent this reaction.

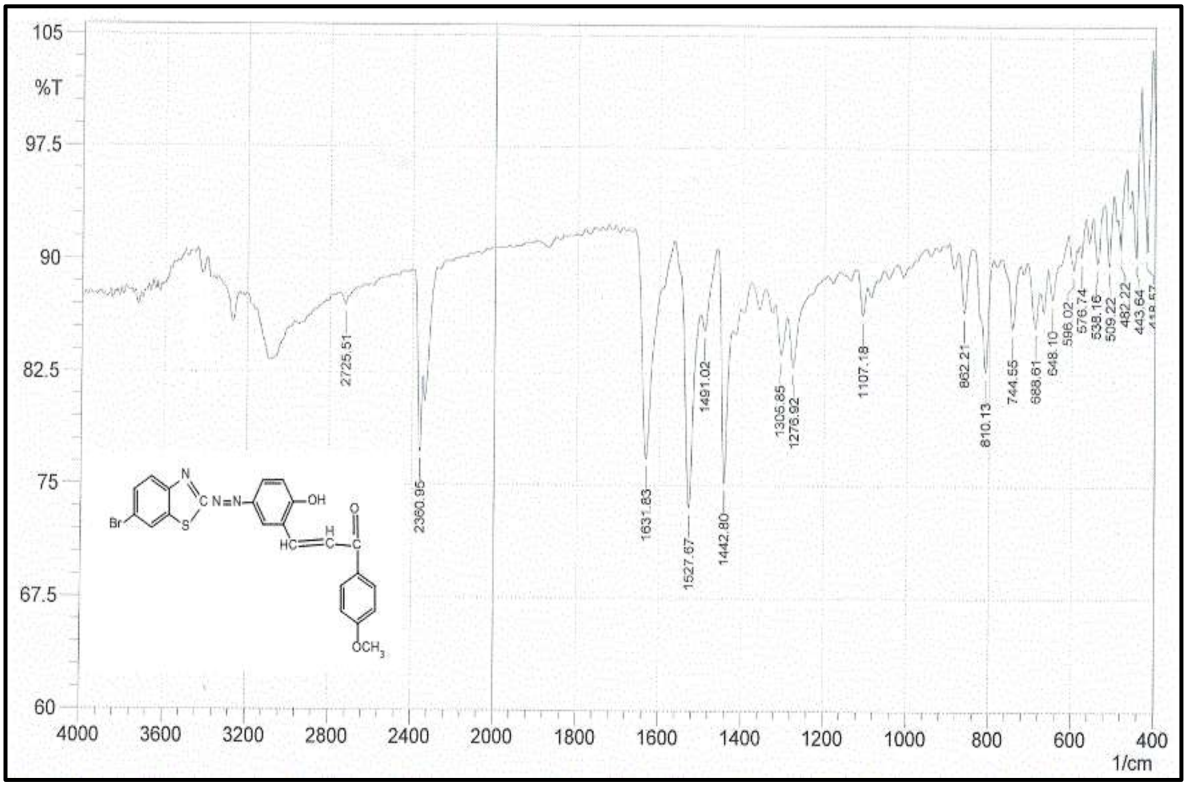


Figure 11: The FT-IR spectrum of compound 3A

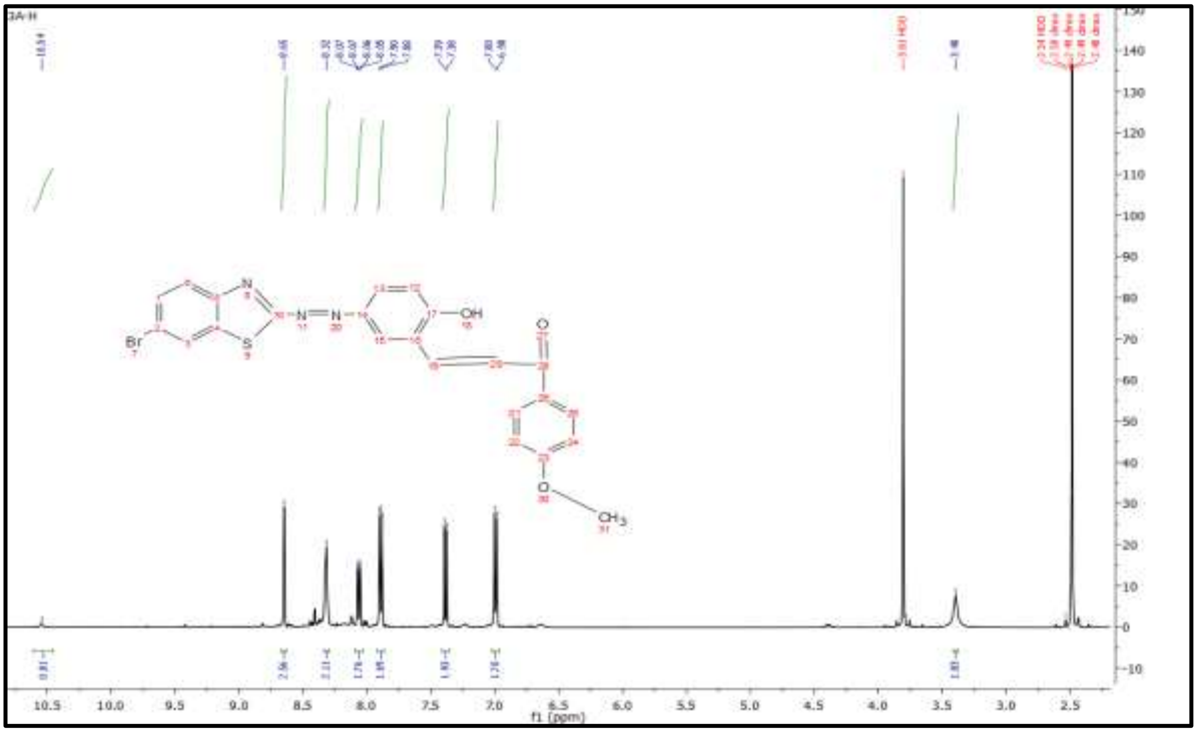


Figure 12: The ^{1}H-NMRspectrum of compound 3A

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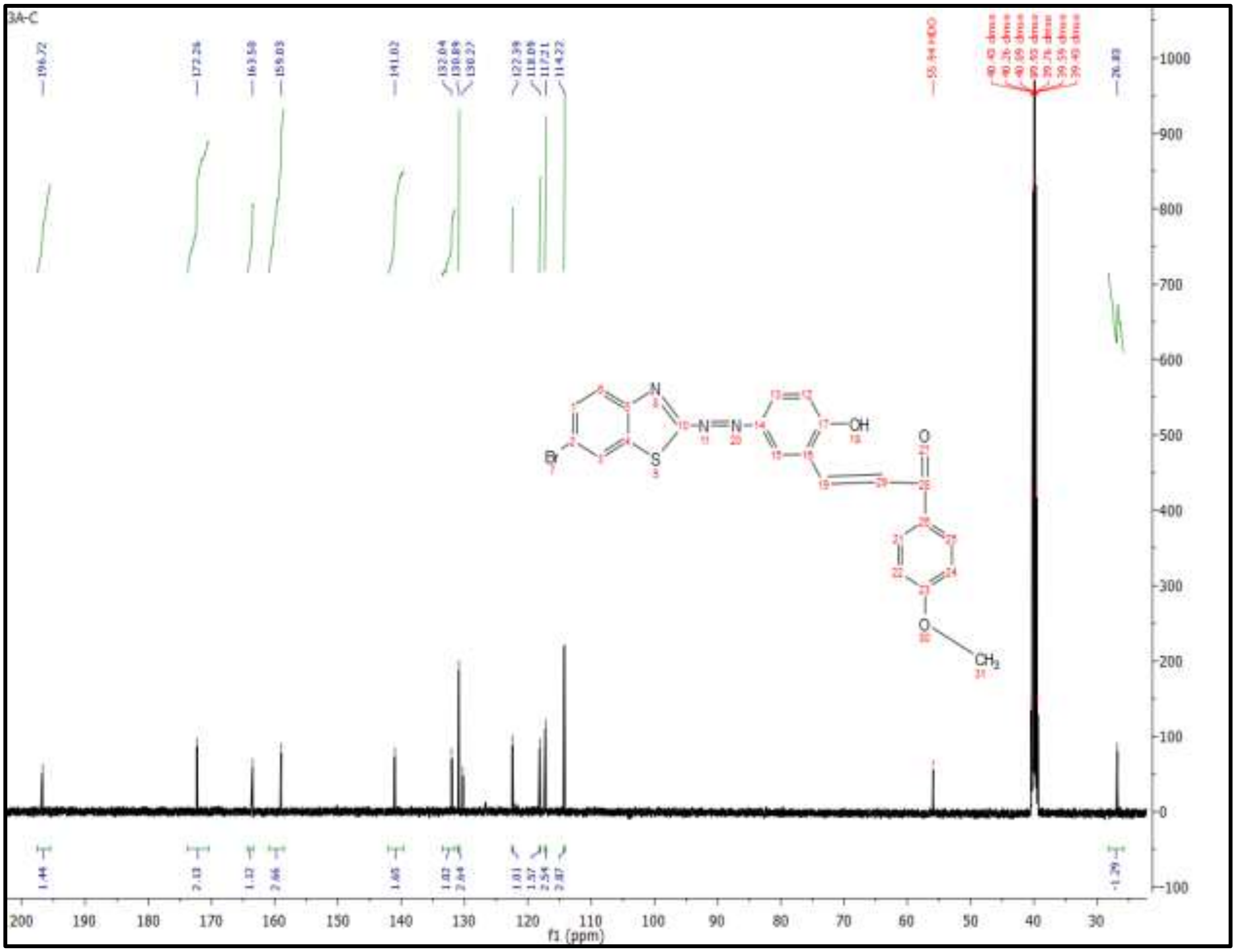

Figure 13: The ^{13}C-NMRspectrum of compound 3A

4- Preparation of additive 4A; Figure (14),The spectrum of the FT-IR of the derivative (4 A) showed; The peak appearance at (3330) cm $^{-1}$ to the stretching (OH), the peak at (3095) cm $^{-1}$ returns to the stretching(=CH)aromatic in the ring, the peak at frequency (1301) cm $^{-1}$ is due to (C-H $_{\text{methyl}}$), and the band at (650) cm $^{-1}$ indicates the presence of bromine in the Thiazole ring and the frequency band at (1600) cm $^{-1}$of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1444, 1527) cm $^{-1}$.**Figure (15),**The spectrum of ^{1}H-NMR showed; Single at (9.8) ppm is return to the proton of phenolic hydroxyl group of the ring, the other single at the displacement (3.4) ppm of the (N-Methyl), and singlet at displacement (1.9) ppm return to (CH_3), singlet at displacement (6.4) ppm return to (CH=N), and another multiple signal at (7.2-7.8) ppm of the protons aromatic group. **Figure (16),** The spectrum of ^{13}C-NMR was also showed; Signals at (133) ppm return to C_5, signal at (128) ppm return to $C_4$and signal at (152.09) ppm return to C_{17}, signal also at displacement (21.2) ppm return to C_{29},signal at (167.2) ppm return to C_{16},signal at (127) ppm return to C_{10}signals at displacement at (112-125) ppm returns to aromatic carbon.. **Equation (6),** represent this reaction.

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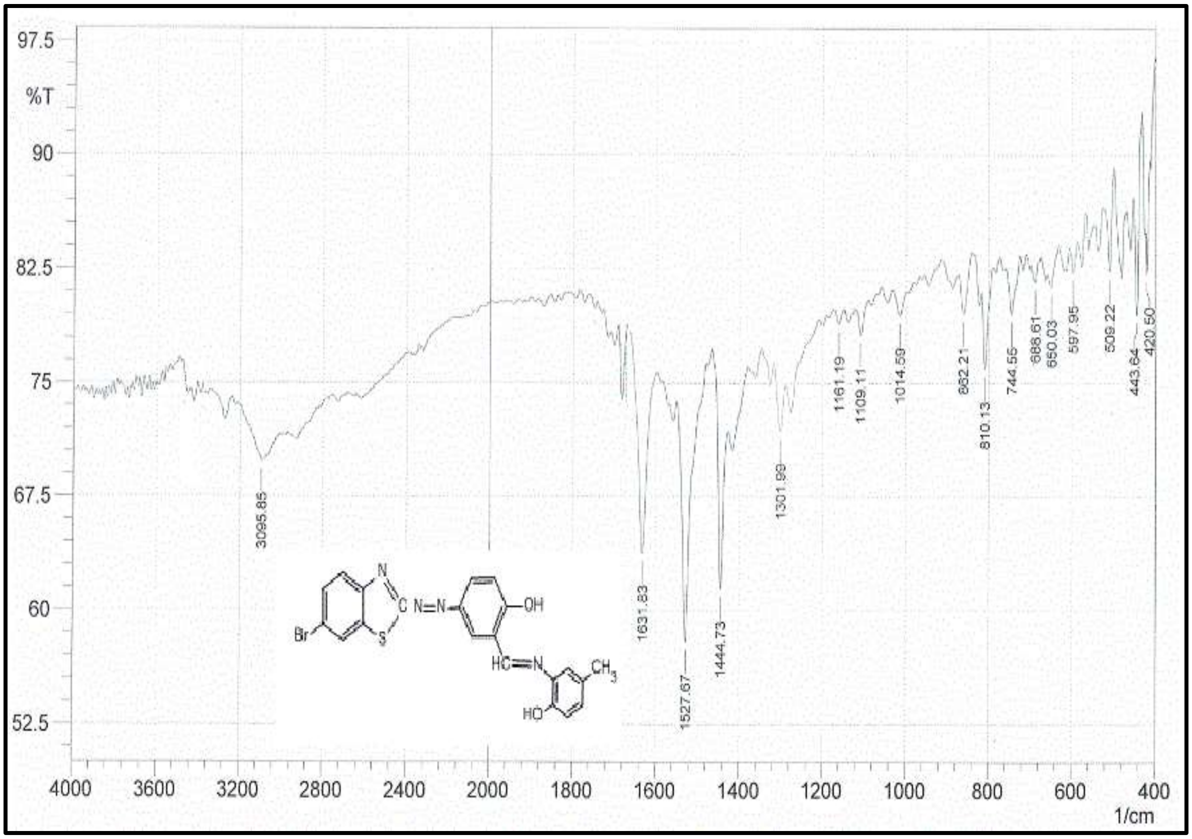

Figure 14: The FT-IR spectrum of compound 4A

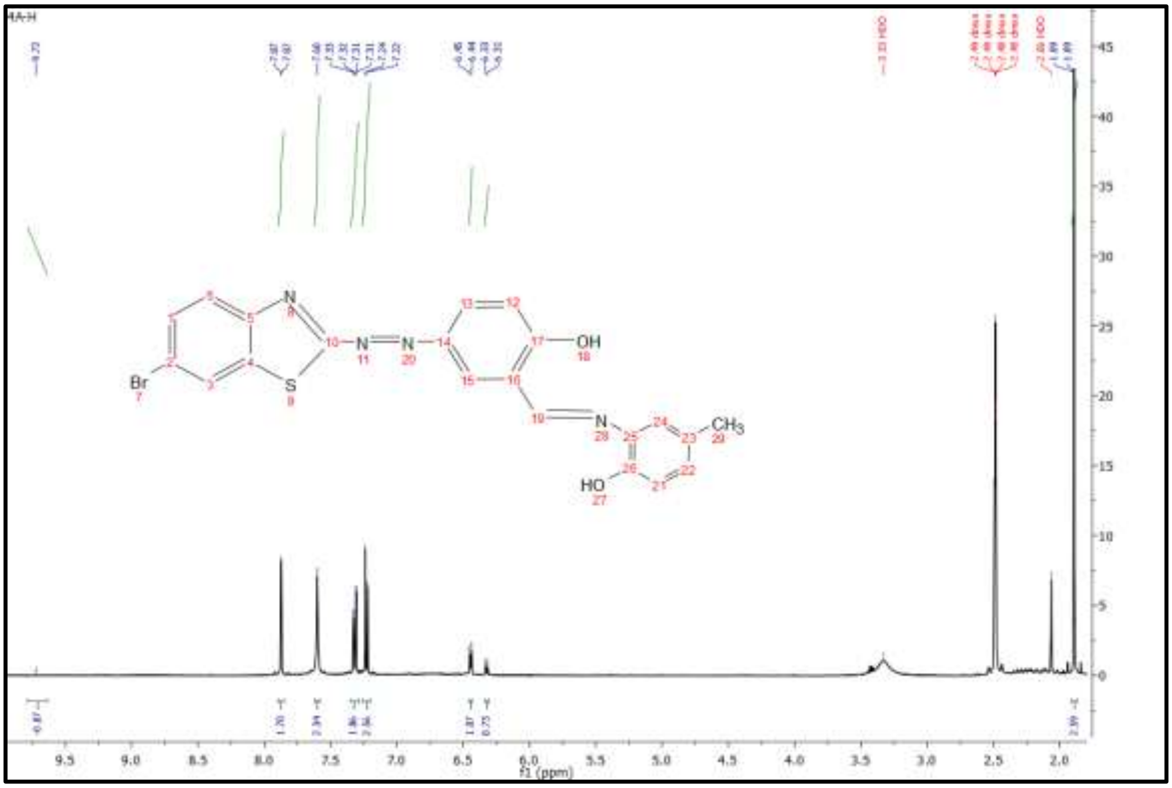

Figure 15: The ^{1}H-NMRspectrum of compound 4A

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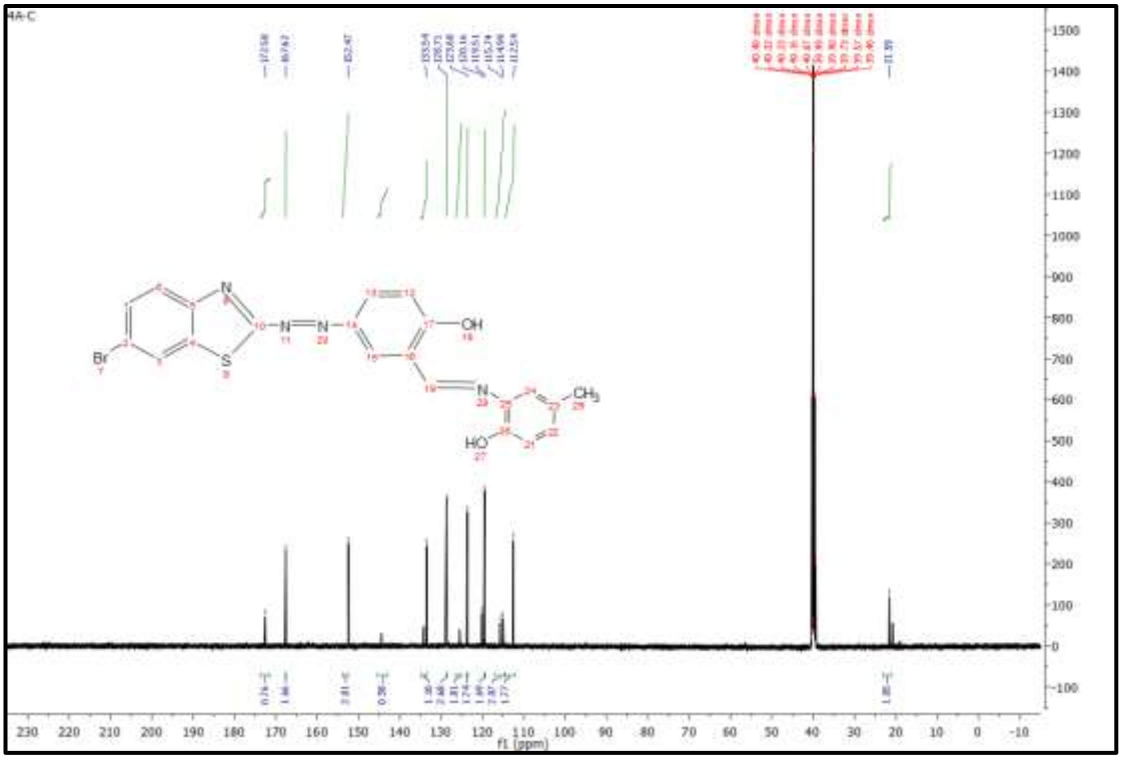
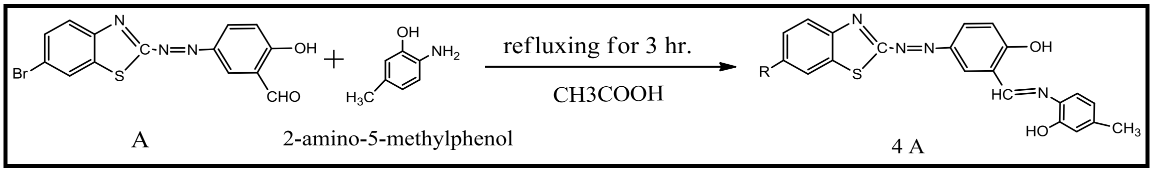


Figure 16: The ^{13}C-NMRspectrum of compound 4A



Equation 6: Synthesis ofcompound4A (shiff base)

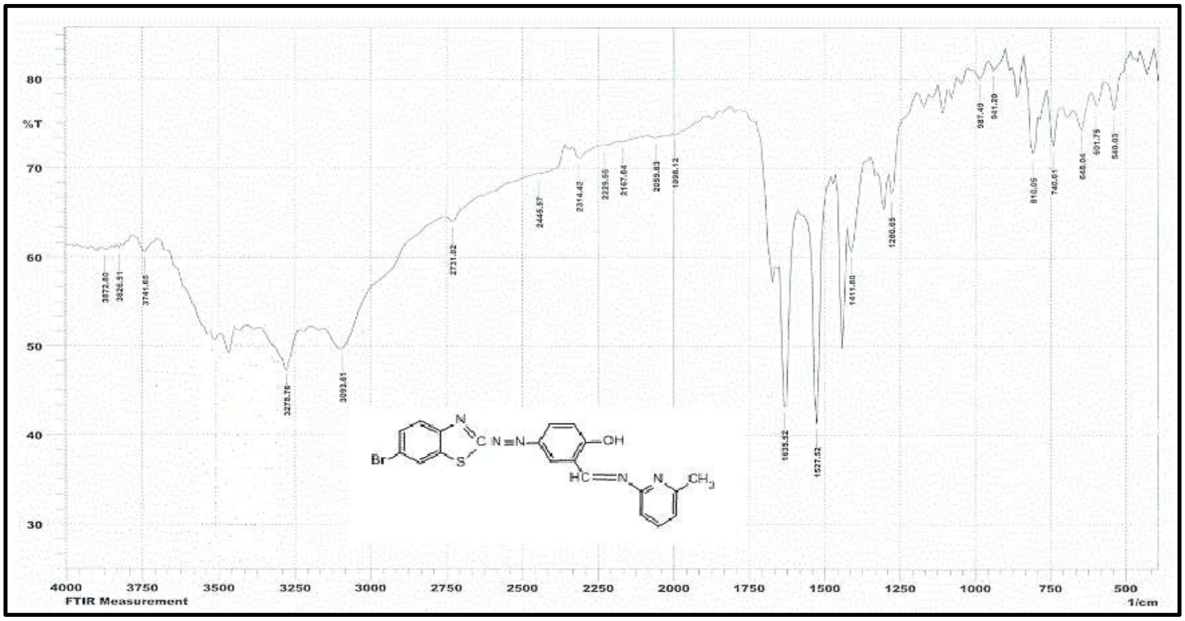


Figure 17: The FT-IR spectrum of compound 5A

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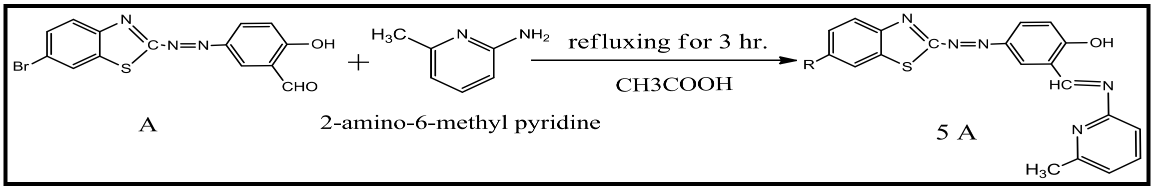
5- Preparation of additive 5A; Figure (17), The spectrum of the FT-IR of the derivative (5 A) showed; The peak appearance at (3278) cm^{-1} to the stretching (OH), the peak at (3093) cm $^{-1}$ returns to the stretching(=CH)aromatic in the ring, the peak at frequency (1280) cm $^{-1}$ is due to (C-H $_{\text{methyl}}$), and the band at (648) cm $^{-1}$ indicates the presence of bromine in the Thiazole ring and the frequency band at (1635) cm $^{-1}$of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1411, 1425) cm $^{-1}$.**Equation (7),** represent this reaction.



Equation 7: Synthesis of compound5A (shiff base)

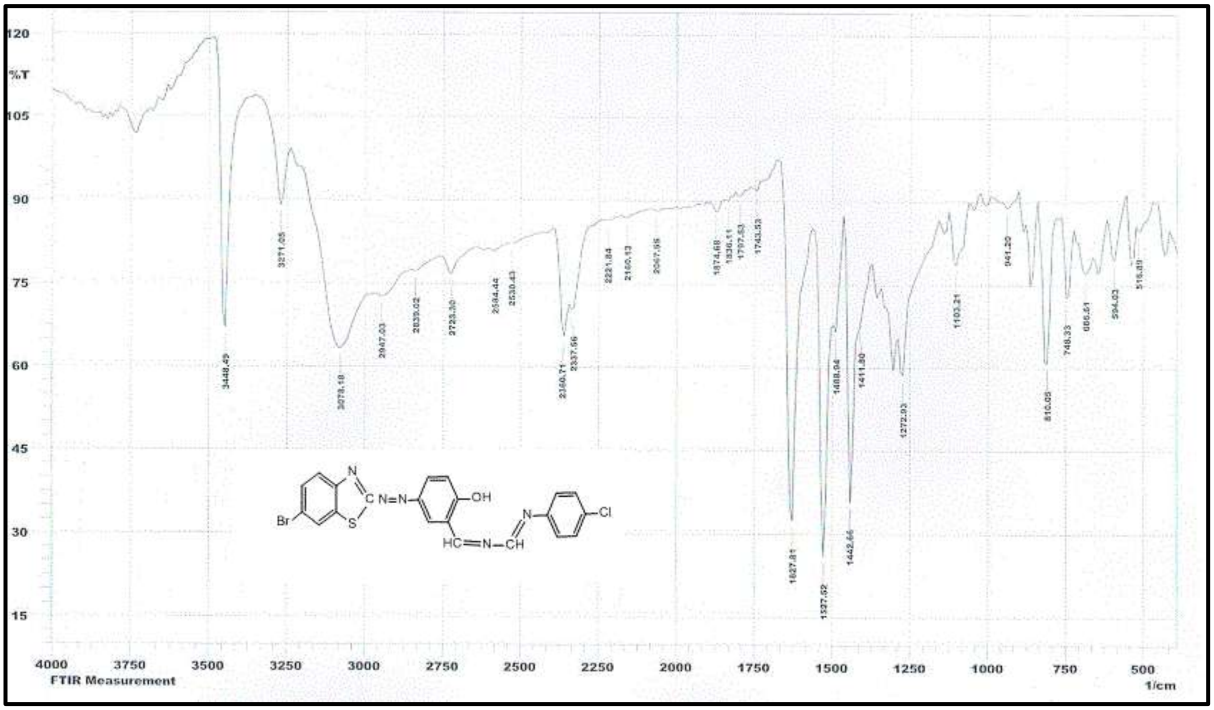
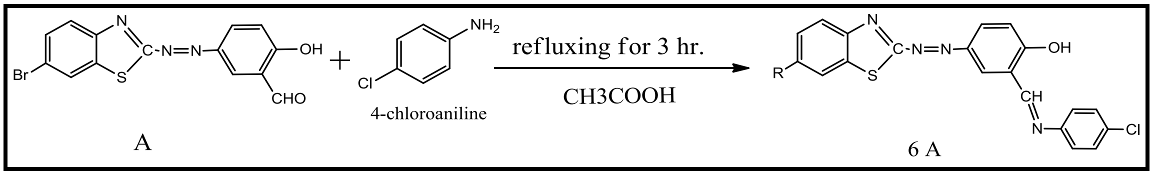


Figure 18: The FT-IR spectrum of compound 6A

6- Preparation of additive 6A; Figure (18),The spectrum of the FT-IR of the derivative (6 A) showed; The peak appearance at (3448) cm $^{-1}$ to the stretching (OH), the peak at (3078) cm $^{-1}$ returns to the stretching(=CH)aromatic in the ring, and the band at (748) cm $^{-1}$ indicates the presence of bromine in the Thiazole ring, the peak at (810) cm $^{-1}$ returns to the(C-Cl), and the frequency band at (1627) cm $^{-1}$of the (C=N), we also note that there are two peaks due to the presence of the azo group at (1442, 1537) cm $^{-1}$.**Equation (8),** represent this reaction.



Equation 8: Synthesis of compound6A (shiff base)

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7-Measurement of LOI using ASTM: D -2863: The limiting oxygen Index (LOI) for unsaturated polyester resin without additives is (20.4) [18] and for epoxy resin without additives is(19.7) [19]; **Tables (2&3) and Figures (19&20)** respectively, indicated that, Oxygen concentration required to support a candle–like in unsaturated polyester and epoxy resins samples were increased with increasing the weight percentage of additives. The efficiency of additives studied was in the following order:

6A > 5A > 4A > 1A > 3A > 2A > A

Table 1: Physical properties of synthesis compound

No.	**Compound**	**M.wt $g.mol^{-1}$**	**m.p C^{o}**	**Yield %**	**Color**	**R_f**
1	***6-bromobenzothiazol-2-amine***	697.10	203	73	Yellow	0.43
2	**A**	362.2	219.8	73	Yellow	
3	**1A**	510.36	140.5	78	Dark brown	0.38
4	**2A**	480.3	solution	69	Black	0.42
5	**3A**	494.36	134.5	67	Dark brown	0.47
6	**4A**	467.34	132	90	Yellow	0.44
7	**5A**	452.33	133	88	Brown	0.41
8	**6A**	498.78	137	85	Dark brown	0.39

Table (2): (LOI) for unsaturated polyester resin with group A of additives

LOI / Additives	LOI %					
	Non	0.1	0.15	0.2	0.25	0.3
A	20.60	21.15	21.71	22.31	22.87	23.25
1A	20.60	22.42	23.13	23.92	24.63	25.10
2A	20.60	21.84	22.70	23.16	23.87	24.32
3A	20.60	21.96	22.88	23.35	23.96	24.45
4A	20.60	23.40	24.19	24.95	25.76	26.16
5A	20.60	23.71	24.57	25.47	26.31	26.73
6A	20.60	23.96	24.82	25.80	26.94	27.36

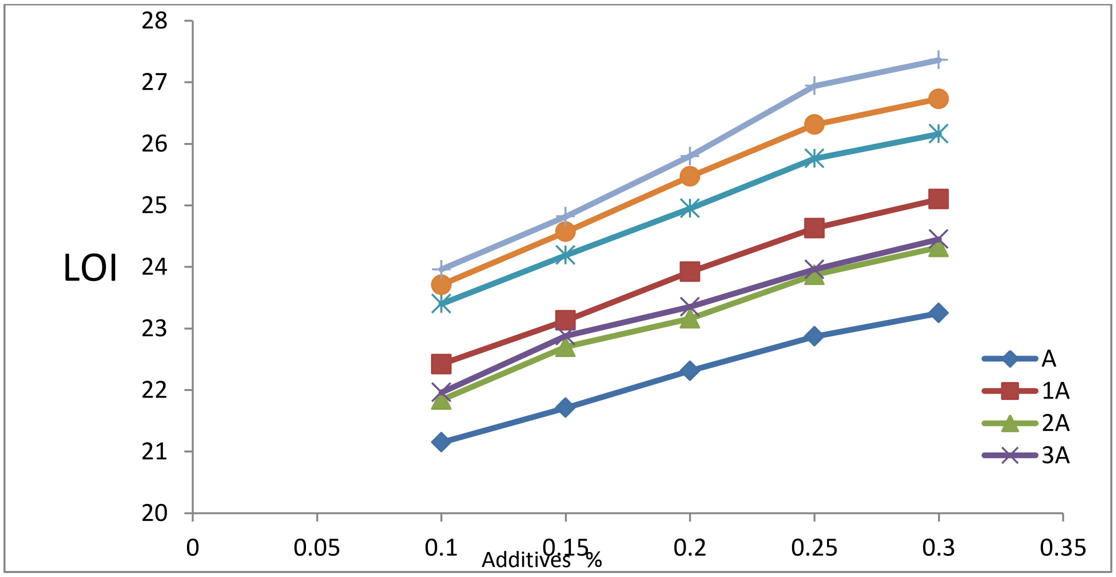

Figure 19: (LOI) for unsaturated polyester resin with group A of additives

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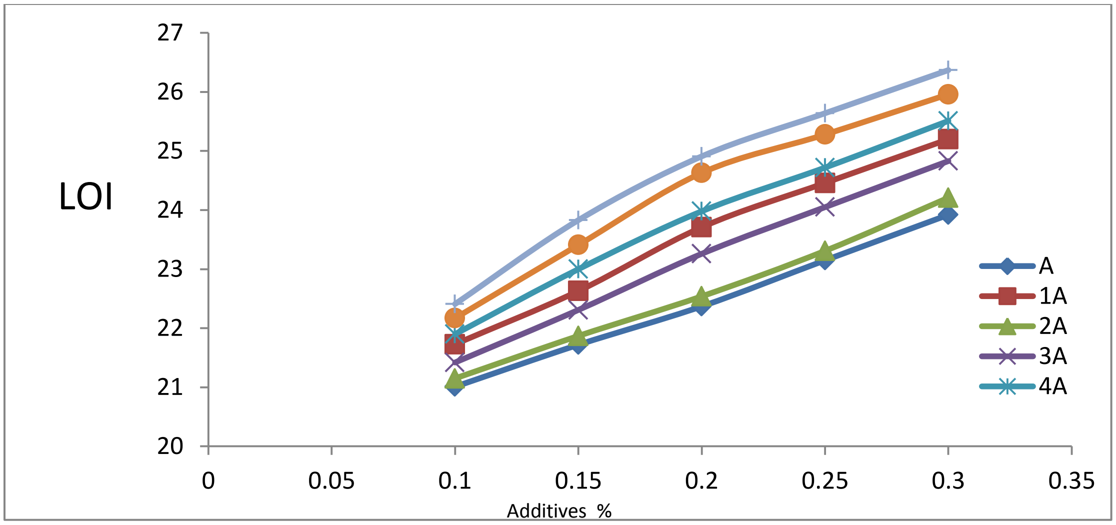

Figure 20: (LOI) for epoxy resin with group A of additives

2-Measurement of rate of burning (R.B) using ASTM: D-635

The rate of burning **(R.B)**for unsaturated polyester resin without additives is (1.44) [20] and for epoxy resin without additives is(1.95) [21]**;**Results obtained from these tests showed that the rate of burning (R.B) of these resins with additives are of inversely proportional with the percentage weight of additives, as indicated in **Tables (4&5), Figures (21&22)** respectively, showed the flame speed curves of flame retardation for the resins. The results obtained from these measurements correspond to the results obtained from the limiting oxygen index measurements. The efficiency of additives studied was in the following order:

6A > 5A > 4A > 1A > 3A > 2A > A

Table 3: (LOI) for epoxy resin with group A of additives

LOI Additives	LOI %					
	Non	0.1	0.15	0.2	0.25	0.3
A	19.54	21.01	21.72	22.37	23.15	23.92
1A	19.54	21.73	22.63	23.71	24.46	25.20
2A	19.54	21.15	21.87	22.54	23.31	24.21
3A	19.54	21.42	22.31	23.26	24.05	24.83
4A	19.54	21.90	23.00	23.98	24.72	25.51
5A	19.54	22.17	23.41	24.63	25.28	25.96
6A	19.54	22.41	23.83	24.91	25.64	26.37

Table 4: Rate of burning (R.B) for unsaturated polyester resin with group A ofadditives

Tests	**Additives %**						**Additives**
	Non	**0.1**	**0.15**	**0.2**	**0.25**	**0.3**	
AEB **(cm.)**	10	10	10	8.3	7.5	7.0	**A**
	10	8.5	7.0	4.5	-	-	**1A**
	10	10	10	8.0	6.7	6.1	**2A**
	10	10	10	7.1	5.3	4.7	**3A**
	10	7.1	4.6	-	-	-	**4A**
	10	5.4	-	-	-	-	**5A**
	10	2.2	-	-	-	-	**6A**
ATB	6.92	7.30	7.81	6.85	6.41	6.31	**A**
	6.92	10.24	10.76	11.25	-	-	**1A**
	6.92	8.00	8.33	7.01	6.14	5.92	**2A**
	6.92	8.92	9.52	7.72	6.16	6.61	**3A**
	6.92	9.37	14.83	-	-	-	**4A**

(Min.)	6.92	10.84	-	-	-	-	**5A**
	6.92	12.62	-	-	-	-	**6A**
R.B. **(cm./ Min.)**	1.44	1.36	1.28	1.21	1.17	1.11	**A**
	1.44	0.83	0.65	0.40	-	-	**1A**
	1.44	1.25	1.20	1.14	1.09	1.03	**2A**
	1.44	1.12	1.05	0.92	0.86	0.71	**3A**
	1.44	0.75	0.31	-	-	-	**4A**
	1.44	0.49	-	-	-	-	**5A**
	1.44	0.17	-	-	-	-	**6A**
S.E	-	-	-	-	-	-	**A**
	-	yes	yes	yes	yes	yes	**1A**
	-	-	-	-	-	-	**2A**
	-	-	-	-	-	-	**3A**
	-	yes	yes	yes	yes	yes	**4A**
	-	yes	yes	yes	yes	yes	**5A**
	-	yes	yes	yes	yes	yes	**6A**
N.B	-	-	-	-	-	-	**A**
	-	-	-	-	yes	yes	**1A**
	-	-	-	-	-	-	**2A**
	-	-	-	-	-	-	**3A**
	-	-	-	yes	yes	yes	**4A**
	-	-	yes	yes	yes	yes	**5A**
	-	-	yes	yes	yes	yes	**6A**

Table (5): Rate of burning (R.B) for epoxy resin with group A ofadditives

Tests	**Additives %**						**Additives**
	Non	**0.1**	**0.15**	**0.2**	**0.25**	**0.3**	
AEB **(cm.)**	10	10	10	8.7	8.1	7.5	**A**
	10	9.0	7.6	6.8	5.8	-	**1A**
	10	10	9.0	8.3	7.6	7.0	**2A**
	10	9.3	8.6	8.1	7.5	6.4	**3A**
	10	7.0	5.9	8.0	6.5	-	**4A**
	10	6.0	4.8	-	-	-	**5A**
	10	4.7	3.5	-	-	-	**6A**
ATB **(Min.)**	5.12	5.88	6.13	5.91	6.00	6.15	**A**
	5.12	7.25	6.49	6.53	6.51	-	**1A**
	5.12	6.80	6.47	6.64	6.84	6.93	**2A**
	5.12	7.04	6.99	7.29	7.81	7.11	**3A**
	5.12	6.73	6.14	9.75	10.65	-	**4A**
	5.12	6.74	8.42	-	-	-	**5A**
	5.12	7.46	8.53	-	-	-	**6A**
R.B. **(cm./ Min.)**	1.95	1.70	1.63	1.47	1.35	1.22	**A**
	1.95	1.24	1.17	1.04	0.89	-	**1A**
	1.95	1.47	1.39	1.25	1.11	1.01	**2A**
	1.95	1.32	1.23	1.11	0.96	0.9	**3A**
	1.95	1.04	0.96	0.82	0.61	-	**4A**
	1.95	0.89	0.57	-	-	-	**5A**
	1.95	0.63	0.41	-	-	-	**6A**
S.E	-	-	-	-	-	-	**A**
	-	-	-	-	yes	yes	**1A**
	-	-	-	-	-	-	**2A**
	-	-	-	-	-	-	**3A**
	-	-	-	yes	**yes**	yes	**4A**
	-	-	yes	yes	yes	yes	**5A**
	-	yes	yes	yes	yes	yes	**6A**
N.B	-	-	-	-	-	-	**A**
	-	-	-	-	yes	yes	**1A**
	-	-	-	-	-	-	**2A**
	-	-	-	-	-	-	**3A**
	-	-	-	-	yes	yes	**4A**
	-	-	-	yes	yes	yes	**5A**
	-	-	-	yes	yes	yes	**6A**

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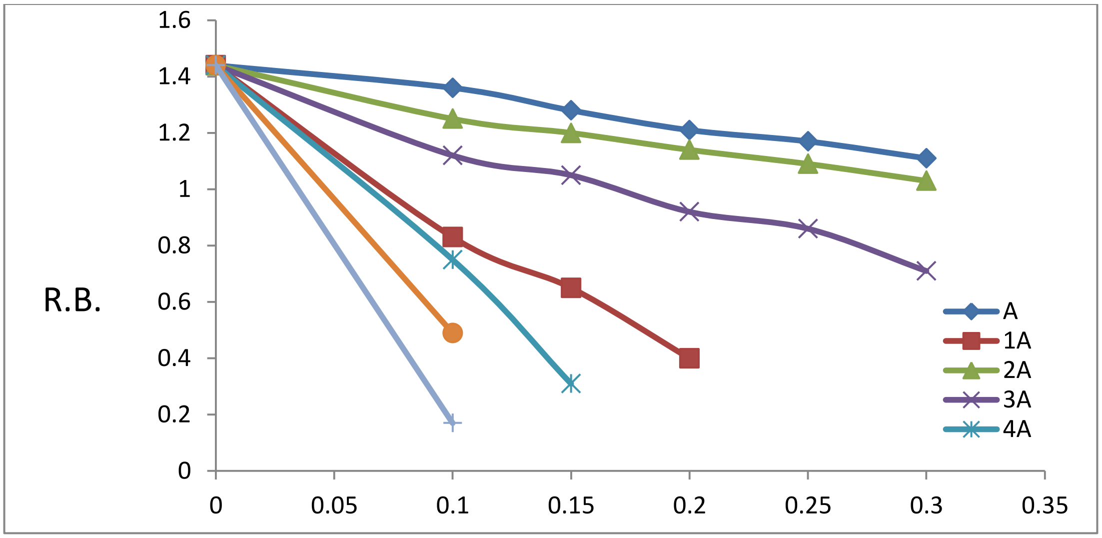

Figure 21: (R.B) for unsaturated polyester resin with group A ofadditives

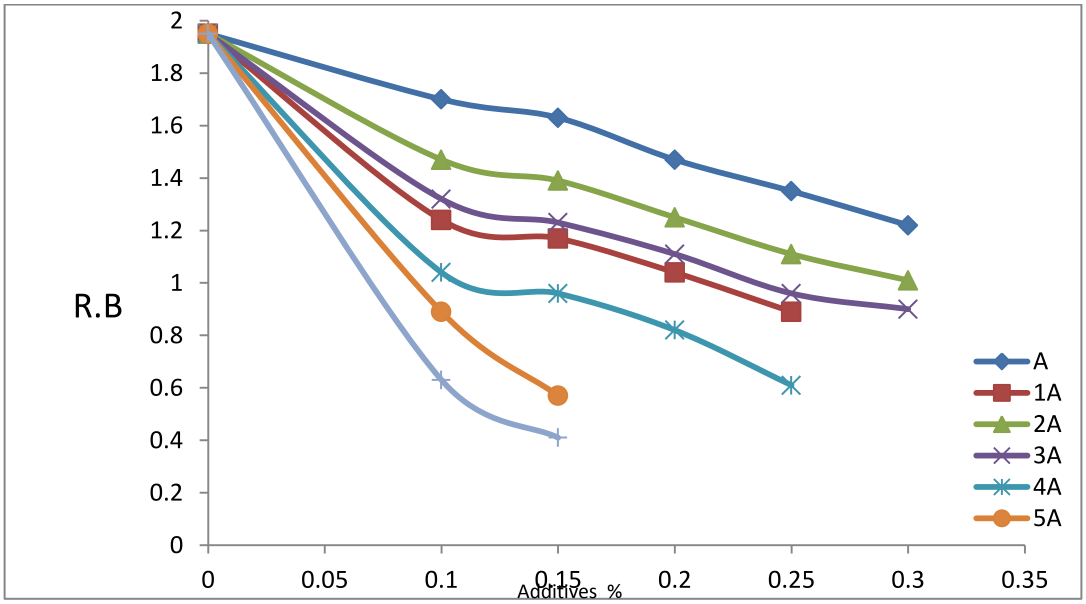

Figure 22: (R.B) for epoxy resin with group A of additives

IV. CONCLUSION

The results obtained can be summarized that, the limiting oxygen index(LOI) increased with increasing the weight percentages of the additives, as well as, the rate of burning (R.B), decreased with increasing the weight percentages of the additives. The flame retarding efficiency of the additives has the following order:

6A > 5A > 4A > 1A > 3A > 2A > A

Apparently, the action of these additives due to by the formation of char as result of removing the hydrogen atoms from the polymer chain with formed the inert compounds. Finally, the combustion products like; free radicals, chare…etc., will form allayer to prevent burning and displacing oxygen that help continues burning of polymers.

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