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The Effect of Azo Moiety on the Electrical Conductivity of Oxadiazole and Triazole Containing Polymers

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Abstract:

Phenolic mercapto oxadiazole and triazole monomers were prepared and then azotized with aniline and Para-phenylene diamine. The prepared monomers have been characterized with FTIR and1HNMR. The six prepared monomers were polymerized by condensation with formaldehyde in a basic medium. The polymer was recognized by FTIR, thermal analysis, and fine elemental analysis (CHN). The results have shown that the presence of azo moiety decreases the thermal stability and the glass transition temperature of the polymers. Also, the oxadiazole polymers are more thermally stable and have higher Tg than the triazole polymers. Doping with different ratios of iodine vapor was carried out on the polymer. The electrical conductivity of the pure and doped polymers was measured by using three probe cells. The results also revealed that the azo moiety increases the conductivity of the pure polymers to some extent (from 1.45534E-11 to 4.69038E-10 ohm-1cm-1 for oxadiazole polymers and from 1.15263E-10 to 3.2680E-10 ohm-1cm-1 for triazole polymers). It was also shown that the electrical conductivity was increased by many orders of magnitude (2-3 orders).by increasing the ratio of the doping.

Key words: oxadiazole, triazole, azo, polymers, electrical conductivity

تأثير مجموعة الازو على التوصيلية الكهربائية للبوليمرات المحتوية على الاوكسادايازول والترايازول

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قسم الكيمياء، كلية العلوم، جامعة الموصل، الموصل، العراق

المستخلص:

تم تحضير مونومرات مركبتوالاوكسادايازول والترايازول الفينولية ومن ثم ازوتتها بالانيلين وبارا- فنيلين ثنائي الامين. درست مواصفات المونومرات المحضرة باستخدام تقنية ^{1}HNMR وFTIR. تم بلمرة المونومرات المحضرة الستة تكثيفيا مع الفورمالديهايد في الوسط القاعدي. درست البوليمرات المحضرة باستخدام تقنية FTIR والتحليلات الحرارية والتحليل الدقيق للعناصر(CHN). اوضحت النتائج ان اضافة مجموعة الازوستقلل من الثباتية الحرارية للبوليمرات وتخفض درجة الانتقال الزجاجي Tg . كما لوحظ ان بوليمرات الاوكسادايازول لها استقرارية حرارية ولها Tg اعلى مما لبوليمرات الترايازول. استعمل بخار اليود لتشويب البوليمرات المحضرة بنسب مختلفة من اليود. تم قياس التوصيلية الكهربائية للبوليمرات النقية والمشوبة باستخدام خلية ثلاثية الاقطاب. اوضحت النتائج ان مجموعة الازو ترفع من قيمة التوصيلية الكهربائية للبوليمرات النقية الى حد ما (من 1.45534E-11 الى

4.69038E-10 اوم$^{-1}$سم$^{-1}$ في بوليمرات الاوكسادايازول بينما في بوليمرات الترايازول من 1.15263E-10 الى 3.2680E-10 اوم$^{-1}$سم$^{-1}$). كما اوضحت النتائج ان التوصيلية الكهربائية للبوليمرات الموشوبة ارتفعت بعدة مراتب (2-3 مرتبة).

الكلمات المفتاحية: اوكسادايازول, ترايازول, ازو, بوليمرات. التوصيلية الكهربائية.

1- Introduction:

The polyconjugation structure in the main chain of the polymer is the major characteristic of the electrically conductive polymers [1]. The essential structure of the conjugated polymers is the presence a of quasi- infinite Π system which extended over the monomer units. The conjugation of Π electrons gives characteristics for the polymer to be highly susceptible to oxidized or reduced chemically or electrically [2]. Oxadiazole and triazole groups can be considered as starting materials used as active heterocyclic compounds in many fields of medicinal, agricultural, and different industrial fields[3]. Oxadiazole and triazole moieties containing polymers represent important types of highly conjugated polymers that have many applications in various fields. The most important applications are electrically conductive polymer synthesis. The electrical conductivity of oxadiazole and triazole containing polymers was studied [4]. It was shown that the electrical conductivity was affected by the chemical structure of the main chain of the polymer. Polymers containing triazole moiety as a side group or in the main chain were used as proton conductivity polymers [5, 6]. In the application of solar cell, it was found that the triazole – based polymers are suitable for bulk hetero junction solar cell, whereby the prepared polymers behaves as a donor [7]. Ayoob *et al.*, proved that the conjugated polymers with diazole ring can behave as n or p type via doping with different dopants [8]. These polymers are used successfully for manufacturing solar cells with acceptable efficiency [9].

On the other hand, the azo group can also enhance the conjugation of the polymer chains. Studies on polyamide [10, 11] and polyester [12] containing azo moiety within the main chain show increase in the electrical conductivity with increasing the number of the azo moieties. Organic polymers with an azo active group were used in lithium ion batteries, whereby the azo moiety can enhance the electrical conductivity by connecting the conjugated structure in one molecular unit and maintaining the electrochemical activity[13]. Polystyrene sulfonate was grafted with azo moiety in order to get photo-illuminated film through the isomerization of the azo group [14].

In the present work, oxadiazole and triazole monomers containing azo moiety as a side group were prepared. Condensation polymers of the prepared monomers were prepared. The electrical conductivity of the pure and doped polymers was investigated.

2- Experimental:

Materials:

Table (1) shows all the used chemicals which were used as received from the sources without any purification except phenylene diamine which was recrystallized from ethanol before used, and the aniline was distilled off twice under vacuum .

Table 1: Used chemicals

	Source	materials
1	Fluka	p-Phenylene diamine
2	Fluka	m- hydroxyl benzoic acid
3	Fluka	Aniline
4	BDH	Hydrazine hydrate
5	Fluka	Potassium hydroxide
6	BDH	Sodium hydroxide
7	Sigma Aldrich	Hydrochloric Acid
8	Analar	Sodium nitrate

9	Scharlau	phosphoric acid
10	Fluka	Con. Sulfuric acid
11	BDH	Iodine
12	BDH	formaldehyde
13	BDH	Dimethyl sulfoxide (DMSO)
14	BDH	Sodium carbonate
15	Fluka	Ethanol Absolute.
16	Fluka	Carbon disulfide

Equipment:

FTIR spectra were accomplished by using (BRUKER F.T.IR Infrared. The spectra are obtained using the powder of the sample.

^{1}HNM was performed by using Bruker Bio GmbH Spectrophotometer (400 MHz) by using DMSO as a solvent.

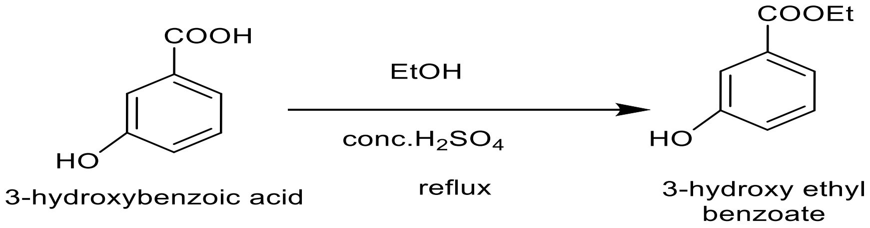
Thermal analyses were measured by Metler, the heating speed of measuring TGA and DTA of the prepared samples was 20^{o}C\min**.**

CHN elemental analysis was measured by using Veri OEL V5.

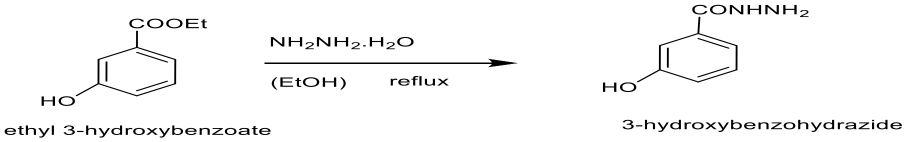
The volume electrical conductivity measurements for the pure and doped polymers were performed by using a lab –mad 3probe Dc cell [15].

Methodology:

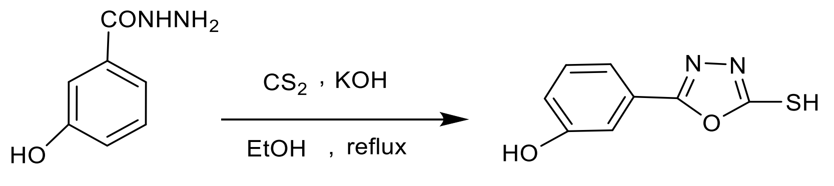
3- hydroxyl ethyl benzoate: was prepared from 3- hydroxyl benzoic acid by using conc. H_2SO_4 as a catalyst. was added and reflux the mixture for a period of 8hrs.. After that, the excess ethanol was distilled off. 30ml of crushed ice was added to the mixture and neutralized by 10% Na_2CO_3. The precipitated product was filtered and dried under vacuum, m.p.= 117^{0}C, yield= 81%.



3- hydroxyl benzohydrazide: 15 ml of hydrazine hydrate was added to 4.2g (0.02mol) of the above prepared ester. The mixture was refluxed for 3hrs, 50ml of ethanol was added to the mixture, and continued reflux for another 3hrs. After completion of the period of reaction, the excess ethanol was distilled off and crushed ice was added to the residue. The precipitated product was filtered and washed with cold ethanol, m.p.= 265-267^{0}C, yield= 85%.



1,3,4- Oxadiazole (M1): 0.05 mole of 3- hydroxyl benzohydrazide was dissolved in 75ml of abs. ethanol, 3g of KOH was added and 3ml of CS_2 was added drop wise with stirring in an ice path. The reaction mixture was refluxed for a period of 10 hrs., the appearance of yellow vapor as H_2S refers to the completion of the reaction. Excess ethanol was distilled of and the product was precipitated by adding crushed ice and acidifying the medium to pH=2 by 1NHCl. The precipitate was filtered and recrystallized from ethanol- water mixed solvent. m.p= 224-226^{0}C, yield = 76% .



3-hydroxybenzohydrazide

3-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol M1

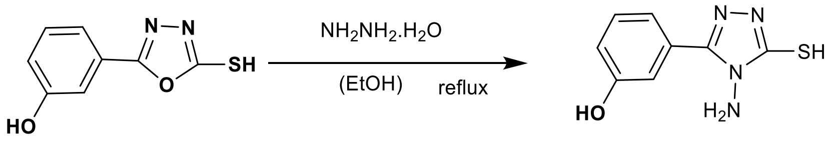
Azotization of M1:

1- with aniline (M2): 0.93g (0.1 mol.) of freshly pure aniline was dissolved in 10ml of conc. HCl at 0-5^{o}C. Aqueous solution of $NaNO_2$ (0.69g, 0.01mol.) was added drop wise at 0-5^{o}C for 30min. (solution A). In another flask, solution B was prepared by dissolving 0.1 mole of M1 in 10ml of 10% NaOH aqueous solution at the temperature 0-5^{o}C. Solution A was added slowly to solution B with maintaining the temperature at 0-5^{o}C. The azo precipitate compound was filtered and dried under vacuum. M.p.= 216- 218^{o}C, yield= 65%.

2- with p- phenylene diamine (M3): the same procedure was followed in preparing M2 except for p-phenylene diamine was used instead of aniline. M.p.= 209-2120C, yield= 60%.

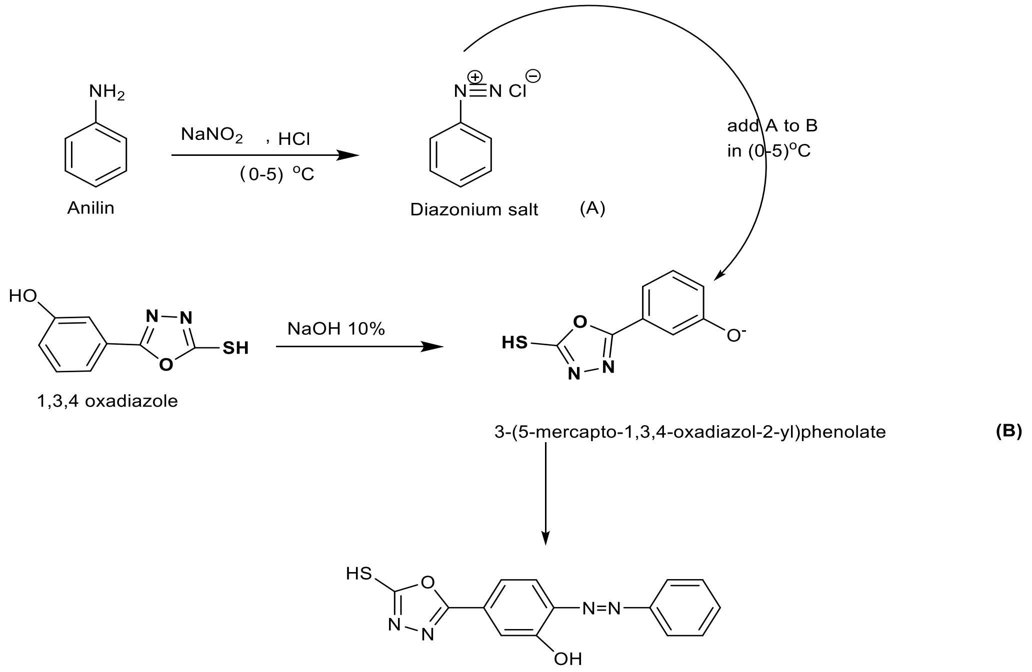
Triazole [4-(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)phenol] M4:

1.9g (0.01mol.) of M1 was mixed with 5ml(0.03 mol.) of hydrazine hydrate and 50ml of abs. ethanol. The mixture was refluxed for a period of 8hrs. The excess ethanol was distilled off, 50ml of crushed ice was added and then the mixture was acidified to pH 2 by 1NHCl. The precipitate was filtered and dried under vacuum. M.p.= 264-266^{0}C, yield= 90%.

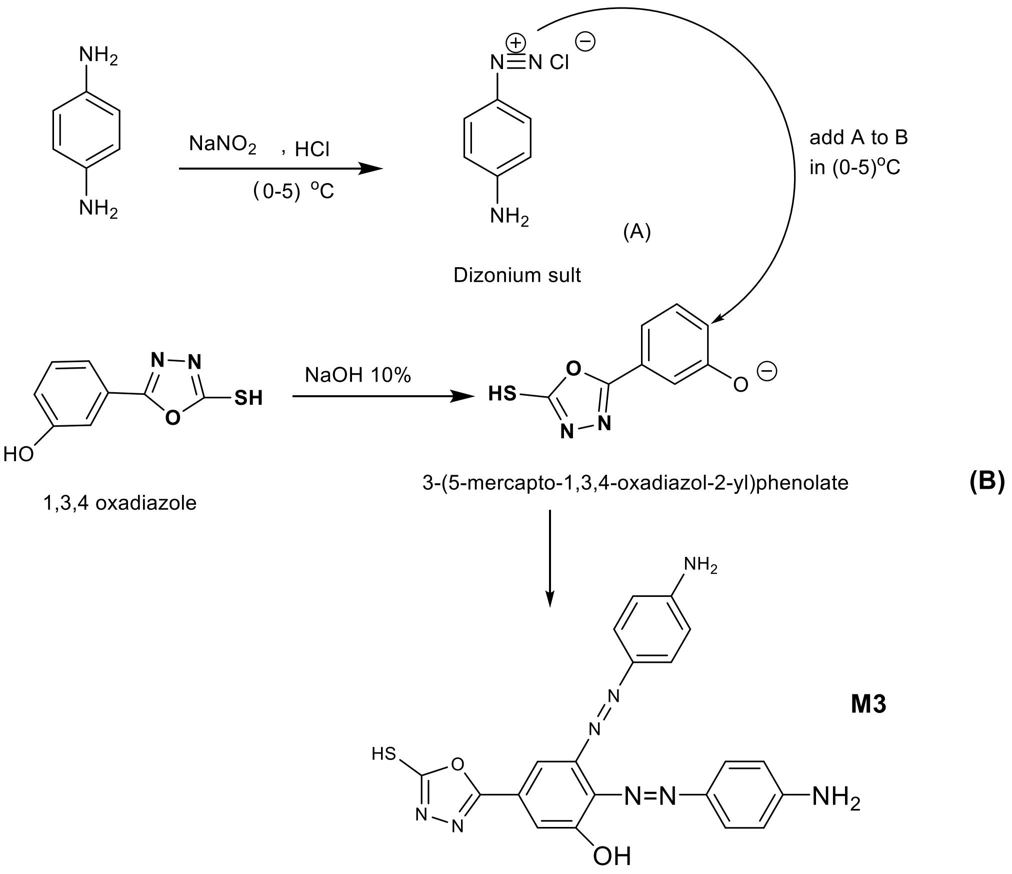


3-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol

3-(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)phenol (M4)

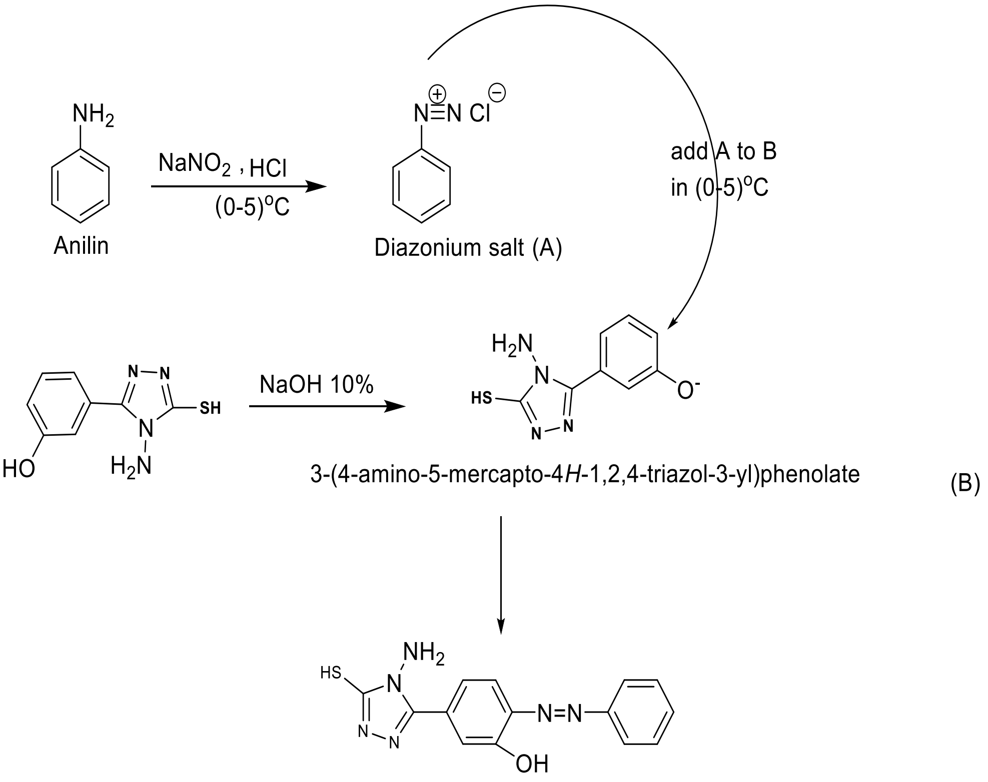


(*E*)-5-(5-mercapto-1,3,4-oxadiazol-2-yl)-2-(phenyldiazenyl)phenol **M2**

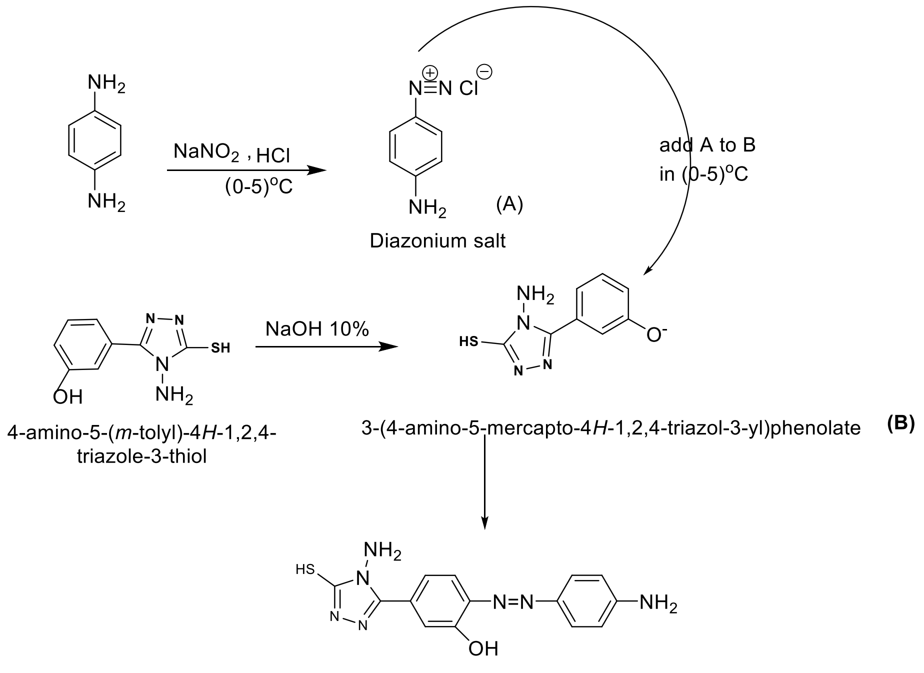


3-((4-aminophenyl)diazenyl)-2-((*E*)-(4-aminophenyl)diazenyl)-5-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol

M5&M6: the same procedure was followed in preparing M2 &M3 by using triazole instead of Oxadiazole. M5 has m.p.= 216-218^{0}C, yield= 55%, while M6 has m.p.= 235-240^{0}C, yield= 66%



5-(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)-2-(phenyldiazenyl)phenol **M5**



5-(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)-2-((4-aminophenyl)diazenyl)phenol **M6**

Polymers preparation: Six condensation polymers were prepared from the oxadiazole and triazole monomer. The general procedure for the preparation of polymers (I,II,III,IV,V and VI) is as follows: 0.01 mole of oxadiazole or triazole monomer was mixed with 25ml of formaldehyde solution (37%) in a round bottom flask. 5% NaOH was used to control the pH of the reaction medium at 9.5-10. The mixture was heated to 95^0C and stirred for 8hrs. After completing the period, the mixture was neutralized with 5%H_3PO_4 to pH= 7.5. the produced polymer was collected, washed with deionized water and dried under vacuum

Doping of polymers: The iodine was used as a dopant. Vapor- phase doping process was followed, whereby the polymer disc has been exposed to the vapor of the iodine in an evacuated tube for different periods of time.

Electrical conductivity measurements : Disc was performed from the pure or doped polymers with 2cm diameter and about 0.5mm thickness under 3-4 ton/cm^2 pressure. Electrical volume conductivity measurements are performed using the standard 3-probe D.C technique according to the ASTM method [15] by using the equation:

σ = Id/AE; where: σ =volume conductivity ($S.cm^{-1}$) , I= the flow current (ampere), d=thickness of the specimen (cm), A=active surface area of the specimen (cm^2) and E=applied voltage (volt).

3- Results and discussion:

Synthetic routes:

Monomers preparation:

The oxadiazole and triazole monomers are prepared with routine work. The characteristic groups of the monomers show a distinctive absorption bands in the FTIR spectra (figures 1-6). The absorption bands of the monomers are shown in Table (2).

Table (2): IR absorptions of the prepared monomers

Monomer	OH	S-H	C=N	C- S	N-N	C-H Aromatic	N=N azo	NH_2
M1	3163	2804	1612	750	1479	1274-1166		
M2	3172	2810	1612	839	1506	1265-1170	1525	
M3	3369	3003	1664	837	1479	1265-1170	1581	3182
M4	3335	2978	1614	840	1514	1263-1182	1489	3184
M5	3255	2962	1614	842	1516	1265-1182	1450	3172
M6	3259		1637	839	1508	1265-1184	1489	3172

Tables (3) and Figures (7-12) indicate the chemical shifts appeared in ^{1}H NMR of the prepared monomers.

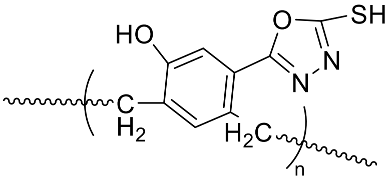
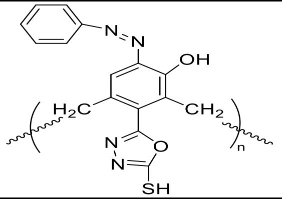
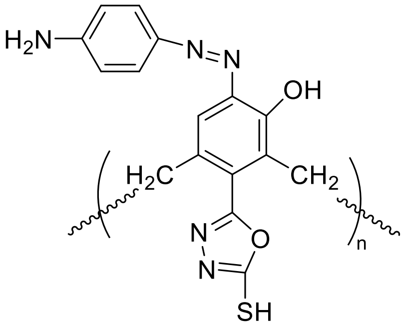
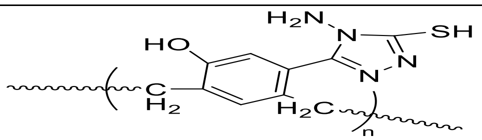
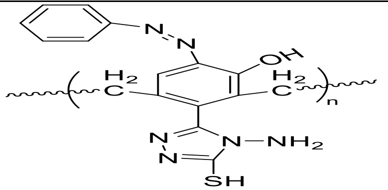
Table 3: Chemical shifts (δ) of ^{1}H NMR bands of the prepared monomers.

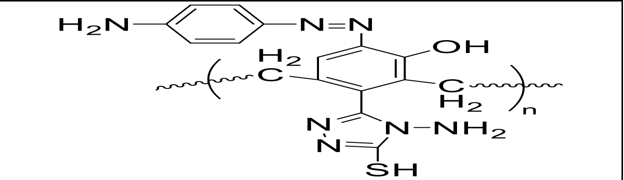
monomer	δSH	δOH	δaromatics	δNH_2
M1	13.77	10.04	7.88- 6.89	
M2	14.53	10.40	7.73-7.71 6.95-6.93	
M3	14.54	10.41	7.73-7.71 6.95-6.93	3.44
M4	13.77	10.04	7.88-6.88	5.74
M5	14.53	10.4	7.98- 7.71	5.74
M6	13.74	10,01	7.91-6.83	6.62

Polymers synthesis:

Six phenoplast polymers were synthesized via condensation polymerization of the oxadiazole and triazole monomers with formaldehyde in a basic medium (Table 4). Whereby the phenol moiety within the monomer structure has two functional groups (two ortho position) that can be condensed with formaldehyde molecule in a basic medium to form the phenol formaldehyde polymers.

Table (4): Chemical structure of the synthesized polymers from condensation between the formaldehyde with the prepared monomers.

polymer	1st monomer	2ndmonomer	Polymer structure
P1	M1	CH_2O	
P2	M2	CH_2O	
P3	M3	CH_2O	
P4	M4	CH_2O	
P5	M5	CH_2O	

P6	M6	CH_2O	

The chemical structure of the prepared polymers was recognized by FTIR spectroscopy (Figures 13-14). In comparison with the spectra of the monomers, the spectra of all polymers show new bands appeared at about 2924- 2942 cm^{-1} which belong to the CH_2 group formed through the polymerization process with formaldehyde to form phenoplast polymers. The other absorption bands of the major monomers also appeared in the spectra of the polymers. The chemical formula of the polymers has been confirmed by fine elemental analysis CHN (Table 5)

Table (5): CHNS elemental analysis of prepared polymers.

Polymer	Calculated				Measured			
	N%	C%	H%	S%	N%	C%	H%	S%
P1	15.800	54.324	3.395	4.527	15.350	54.110	3.115	4.330
P2	11.184	47.934	3.195	6.391	11.090	47.830	3.170	6.200
P3	21.654	51.970	3.518	4.330	21.150	51.650	3.112	3.996
P4	20.790	44.551	3.782	6.052	20.130	43.985	3.200	5.970
P5	22.727	52.108	3.799	4.342	21.860	51.990	3.520	4.100
P6	25.556	50.069	3.911	4.172	24.930	49.792	3.600	3.920

Thermal analysis :

DSC analysis for the pure prepared polymers was performed over 600 ^{0} C under a nitrogen atmosphere(Figures 14and15). Table (6) shows some thermal characteristics of the polymers. The values of Tg can be observed in the range between 366^{0}C to 433.5^{0}C. The results show the effect of the azo moiety on decreasing the Tg as going from P1 to P2 to P3 or from P4 to P5 to P6. The TG analysis was carried out to investigate the thermal stability of the prepared polymers. TGA and DTA curves show two distinct stages for weight loss at 250^{0}C and 600-700^{0}C. The results reveal that the degradation of the polymers is affected by the presence of the azo group which is accelerating the degradation. On the other hand, the oxadiazole polymers (P1,P2, and P3) seem to be more thermally stable than the triazole polymers (P4,P5, and P6) at the two temperatures, whereby the presence of amine (NH_2) as a substituent on the ring of the azo moiety may decrease the stability.

Table (6): Thermal history of the prepared polymers

polymer	Weight loss at 250^{0}C	Weight loss at 600-700^{0}C	TG\ ^{0}C
P1	6.6%	23.7%	433.5
P2	15.06%	55.7%	372.7
P3	13.9%	48.61%	347.7
P4	16.68%	40.97%	380
P5	24.7%	53.5%	362.5
P6	10.9%	28.2%	366.6

Electrical conductivity measurements:

The electrical volume conductivity of the pure prepared polymers was accomplished at room temperature. The numerical values (σ) are listed in Table (7).

Table (7): Electrical conductivity of the purer polymers

Polymer	σ\ ohm^{-1}cm^{-1}	Polymer	σ\ ohm^{-1}cm^{-1}
P1	1.45534E-11	P4	1.15263E-10
P2	4.69038E-10	P5	3.2680E-10
P3	2.13063E-9	P6	2.5633E-10

It is noticed clearly that the chemical structures of the investigated polymers are the dominant factors in affecting the electrical conductivity. On the other hand, it was noticed that the presence of azo moiety will enhance the electrical conductivity, as it on going from P1 to P2 or from P4 to P5. This can be explained by that the azo moieties led to an increase in the polarization of the chain and enhanced the conductivity [11]. In comparison between oxadiazole and triazole polymers (P1 and P4),it was noticed clearly that the presence of NH_2 in triazole will enhance the electrical conductivity with one order, whereby the lone pair of electron of NH_2 group increases the power of conjugation and enhances the conductivity. This idea was confirmed in the conductivity of P3 and P6, whereby there is another NH_2 was added to the phenyl ring of the azo moiety and increased the conductivity.

Effect of Doping on Conductivity:

The delocalization of electrons along the conjugated chains and their jumping from one chain to another through the dopant molecules will represent the process by which charges are transported through the polymeric material [12]. Iodine was carried out as a dopant for the polymers. The iodine was known as an electron donor and enhanced the electrically electronic conductivity [8]. The kinetics of doping exhibit a strong dependence on the doping time. The limitation of the dopants ratio depends on getting the higher values of electrical conductivity or the conductivity become decreasing at higher concentrations of dopant. On the other hand, the polymers may lose their mechanical properties at higher concentrations of the dopants [16]. The values of the volume electrical conductivity of the investigated doped polymers with iodine are shown in tables (8).

Table (8): Electrical conductivity σ (Ω^{-1}cm^{-1}) of the doped polymers with different ratio of iodine.

p	%	σ	%	σ	%	σ
P1	4	9.32E-9	8	6.36E-9		
P2	4	1.24E-9	5	4.46E-9	8	2.20E-8
P3	7	1.51E-9	9	3.40E-9	11	1.08E-8
P4	0.7	1.04E-8	1.0	3.49E-8	1.9	3.32E-8
P5	1	2.64E-9	1.4	1.80E-8		
P6	1.2	1.30E-9	2.1	2.98E-9		

In comparison with Table 7, the electrical conductivity of the doped polymers was increased by many orders of magnitude by doping (2-3 order). The results can be explained in terms of the active moieties (oxadiazole, triazole and azo) can be oxidized with the iodine and forming a charge carriers and enhancing the conductivity. It is well known that the efficiency of the dopant is related to its ability to form some type of complexes that act like charge carriers with the unsaturation units in the polymer chain [17].

4- Conclusions:

1- The oxadiazole and triazole moieties can provide a conjugation for the polymer chains and enhance the electrical conductivity.

2- The azo group as a graft substituent on the chain of oxadiazole and triazole polymers decreases the thermal resistance of the polymers.

3- The azo group can participate in the conjugation of the polymer chains and increase the conductivity to some extent.

4- Doping with iodine vapor increases the electrical conductivity of the polymers by many orders of magnitude.

5. Acknowledgements: The researchers wish to thank the Physical Department\Collage of Science/Mosul University for providing some electrical devices to measure electrical conductivity.

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7- Appendix

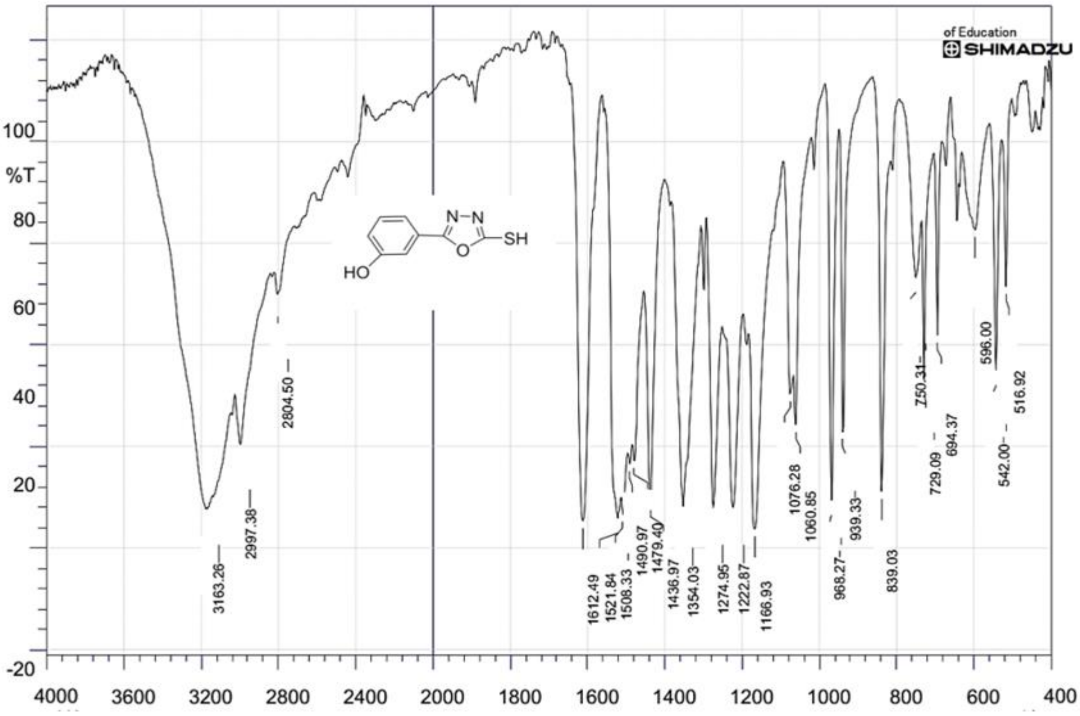


Figure (1): FTIR spectrum of M1

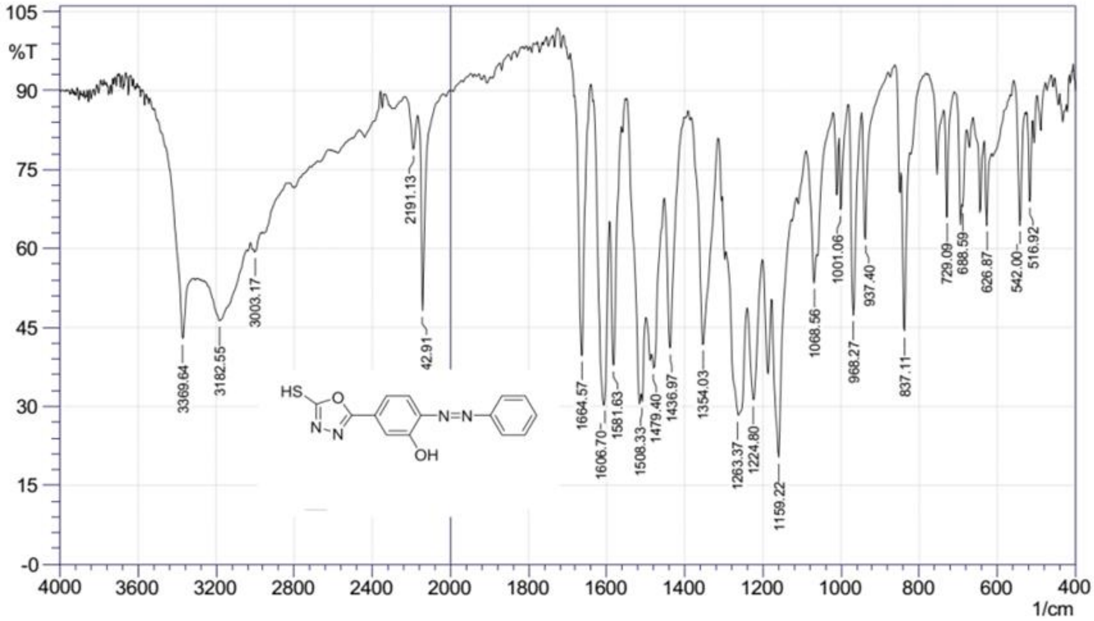


Figure (2): FTIR spectrum of M2

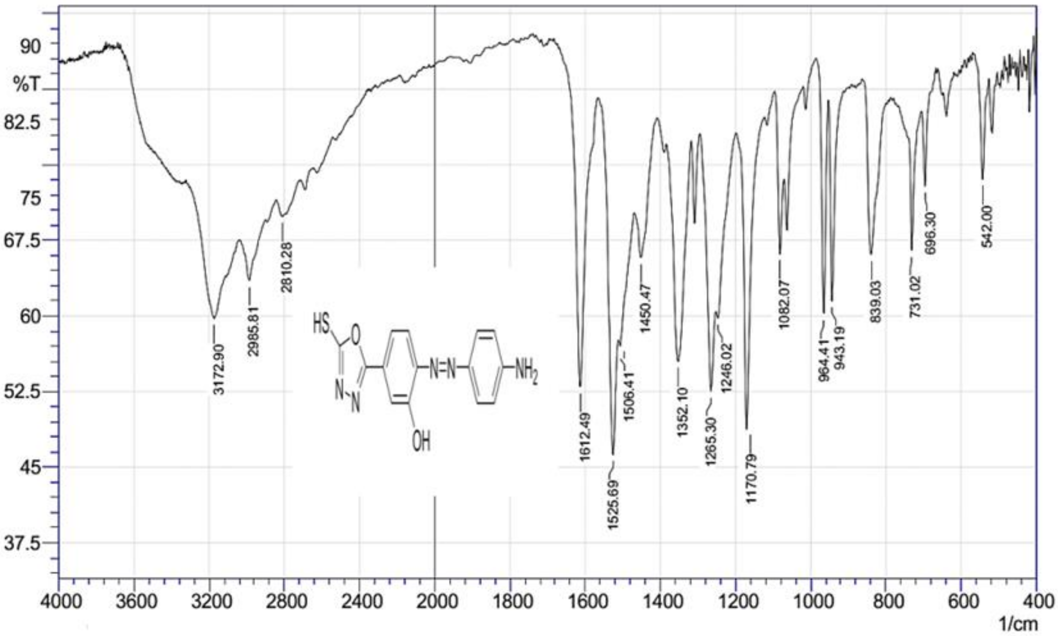


Figure (3): FTIR of monomer M3

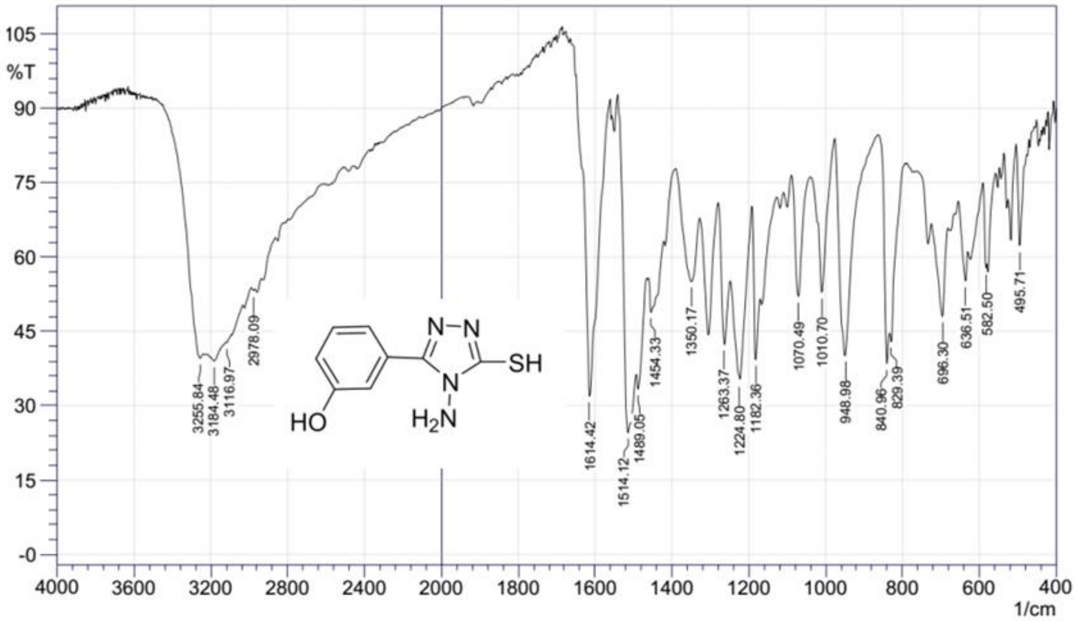


Figure (4): FTIR of monomer M4

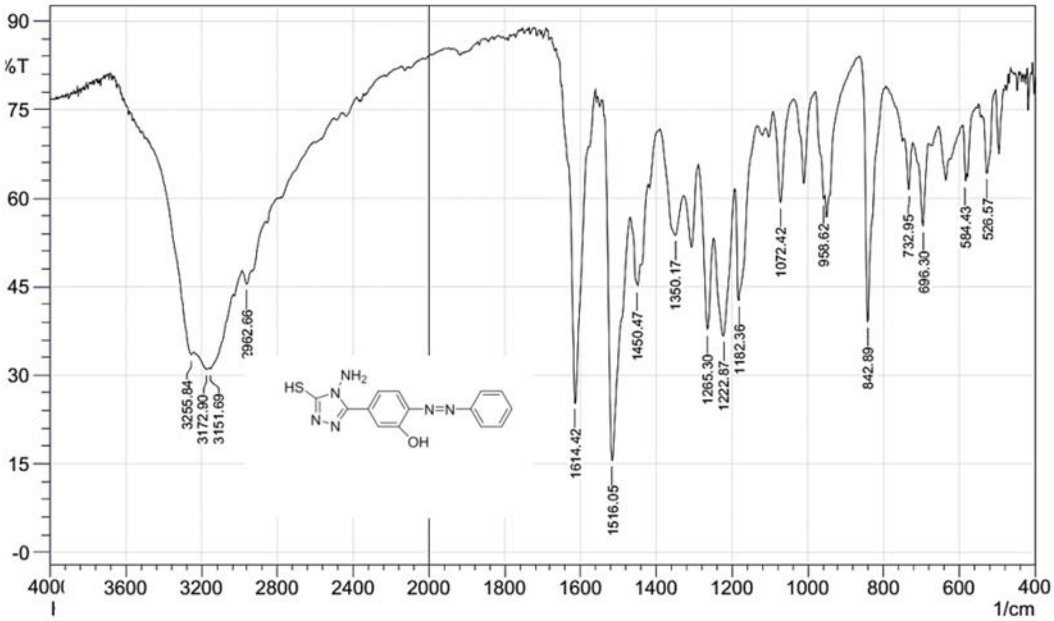


Figure (5): FTIR of monomer M5

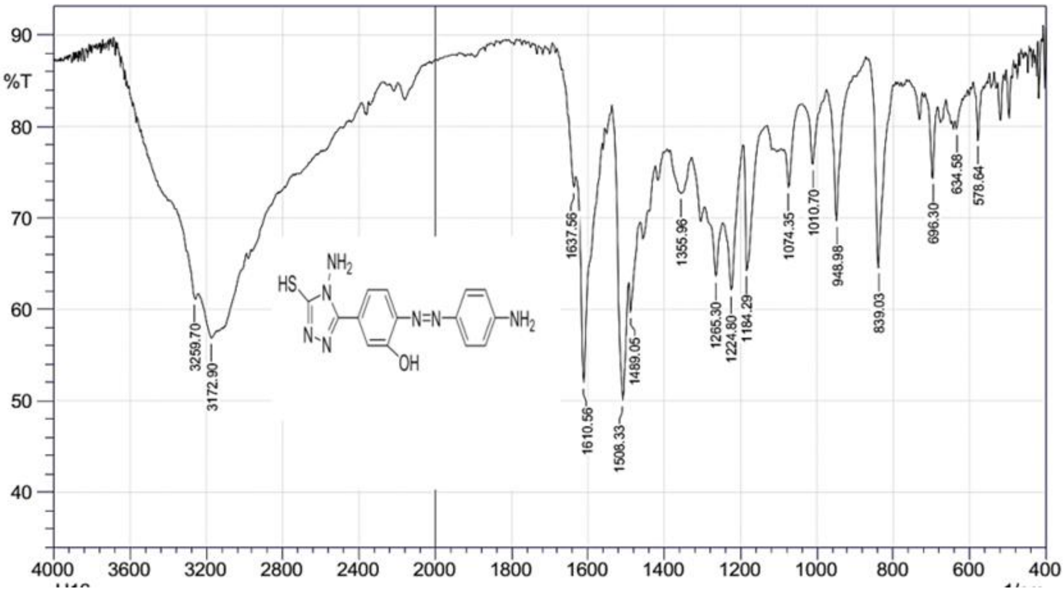


Figure (6): FTIR of monomer M6

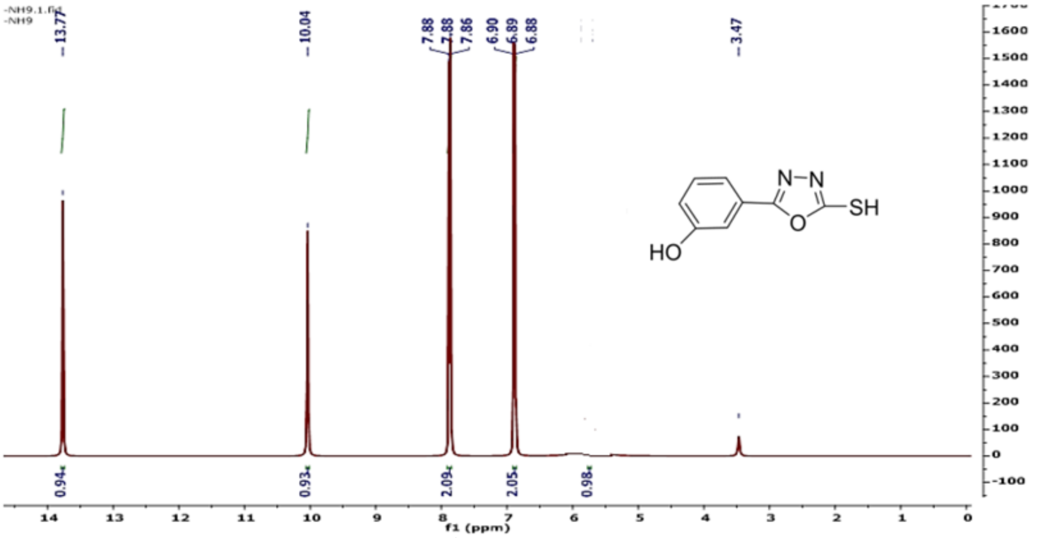
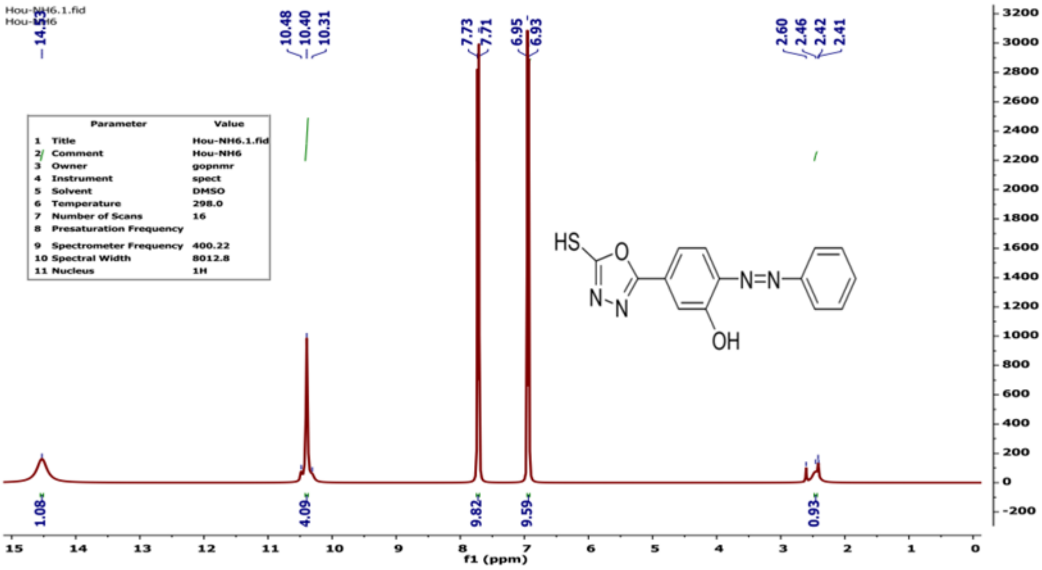


Figure (7): ^{1}HNMR of monomer M1



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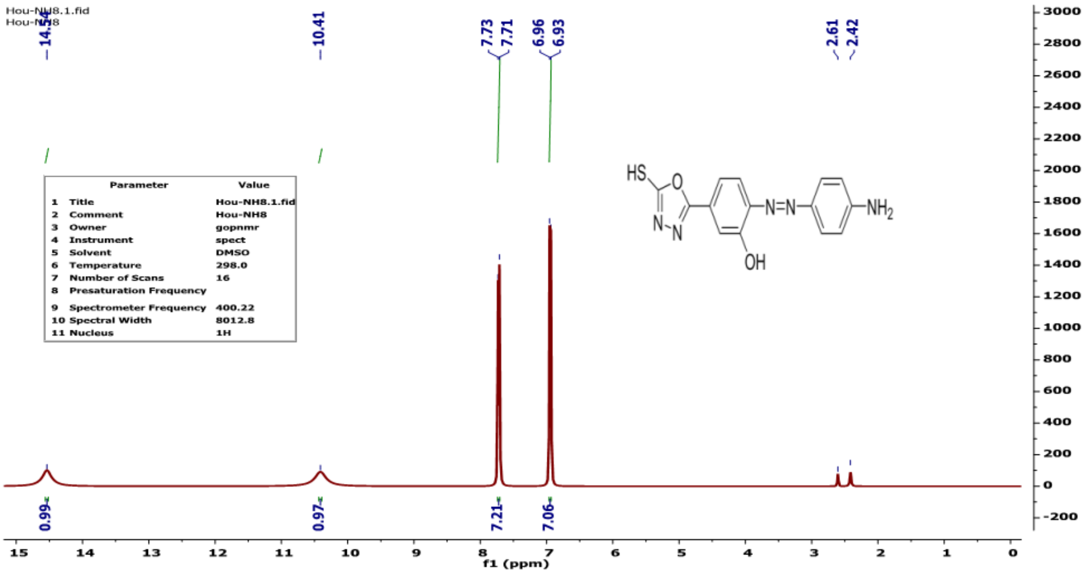
2	Comment	Hou-NH6
3	Owner	gopnmr
4	Instrument	spect
5	Solvent	DMSO
6	Temperature	298.0
7	Number of Scans	16
8	Presaturation Frequency	
9	Spectrometer Frequency	400.22
10	Spectral Width	8012.8
11	Nucleus	1H

Figure (8): ^{1}HNMR of monomer M2



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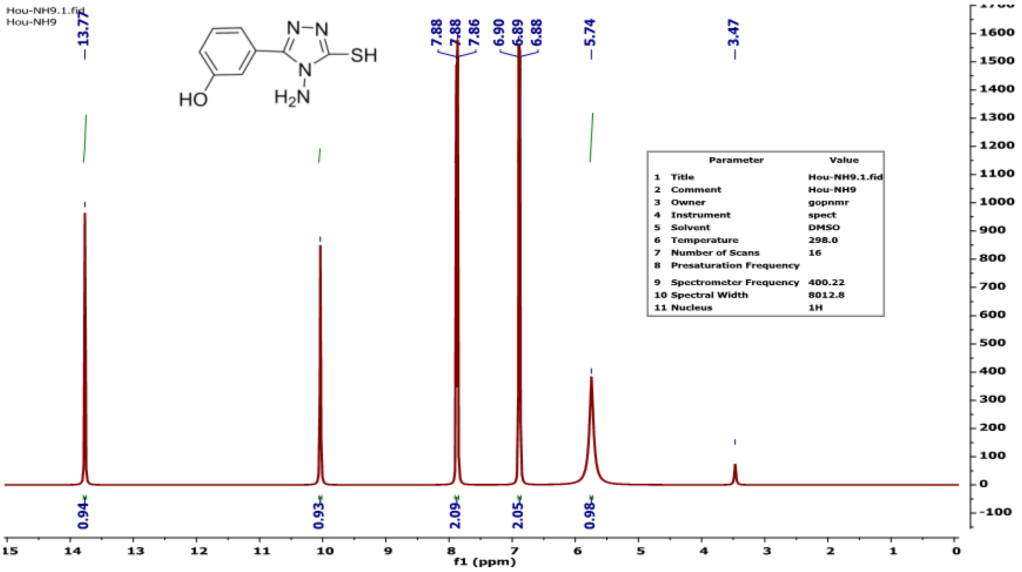
7	Number of Scans	16
8	Presaturation Frequency	
9	Spectrometer Frequency	400.22
10	Spectral Width	8012.8
11	Nucleus	1H

Figure (9): ^{1}HNMR of monomer M3



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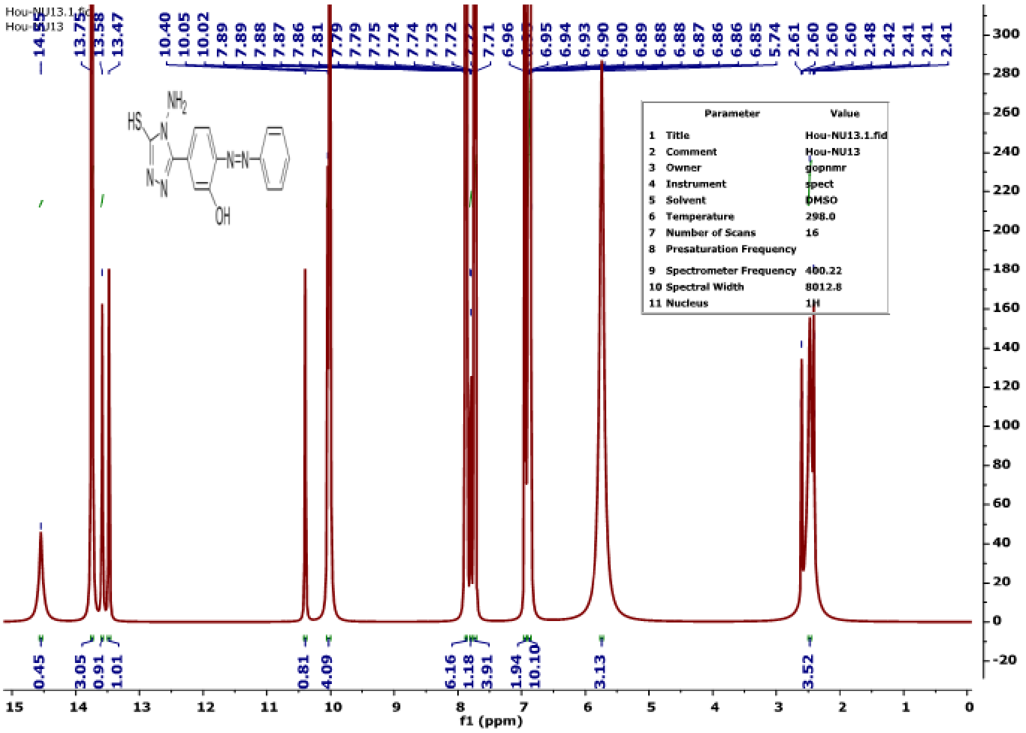
7	Number of Scans	16
8	Presaturation Frequency	
9	Spectrometer Frequency	400.22
10	Spectral Width	8012.8
11	Nucleus	1H

Figuer (10): ^{1}HNMR of monomer M4



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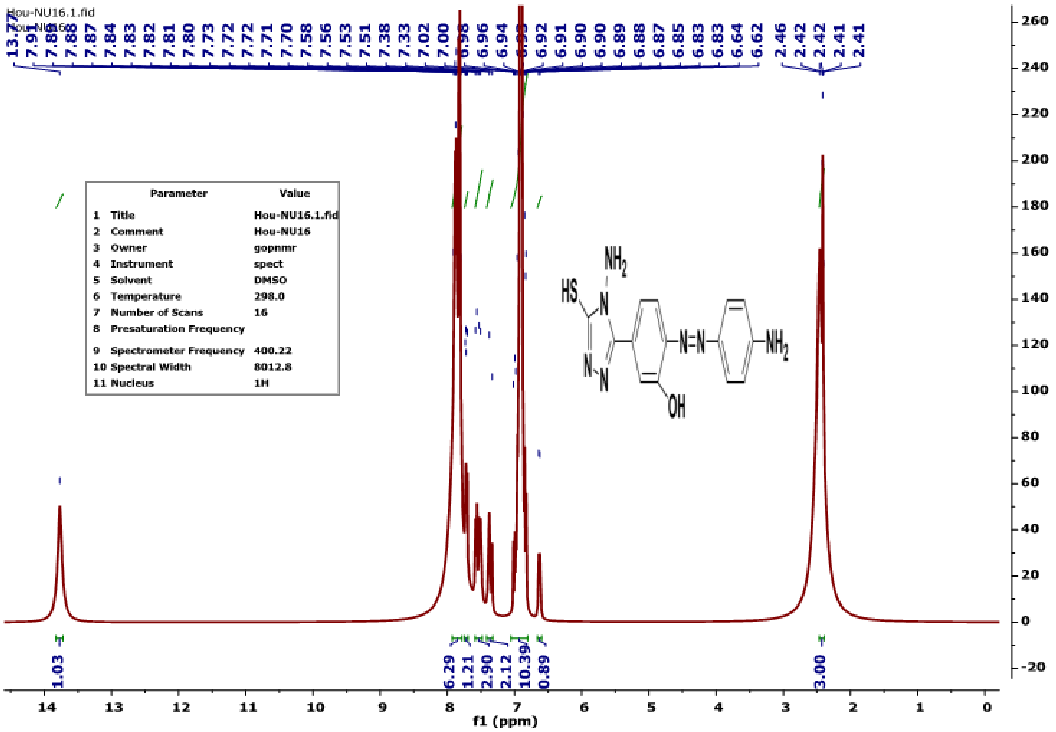
7	Number of Scans	16
8	Presaturation Frequency	
9	Spectrometer Frequency	400.22
10	Spectral Width	8012.8
11	Nucleus	1H

Figure (11): ^{1}HNMR of monomer M5



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7	Number of Scans	16
8	Presaturation Frequency	
9	Spectrometer Frequency	400.22
10	Spectral Width	8012.8
11	Nucleus	1H

Figure (12): ^{1}HNMR of monomer M6

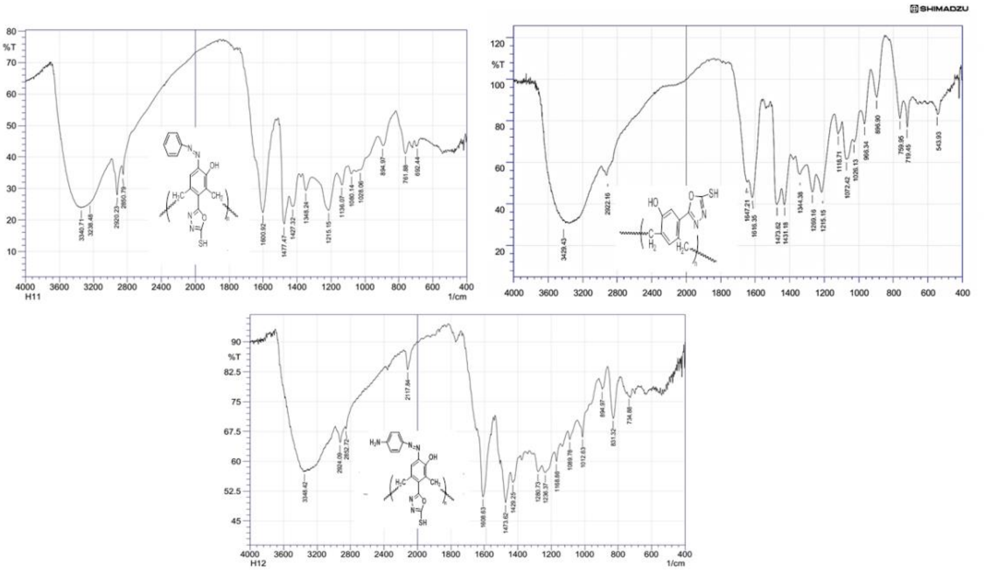


Figure (13):

FTIR of oxadiazole polymers

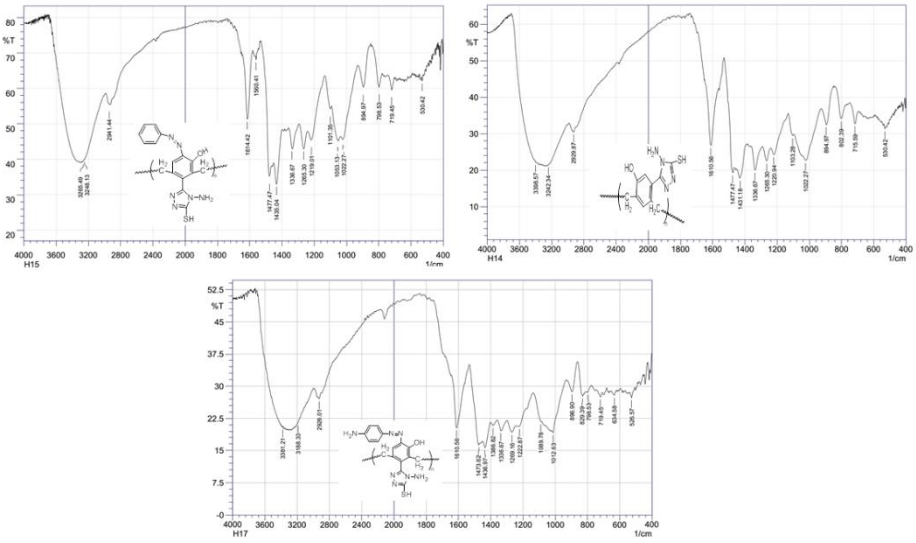


Figure (14):

FTIR of triazole polymers

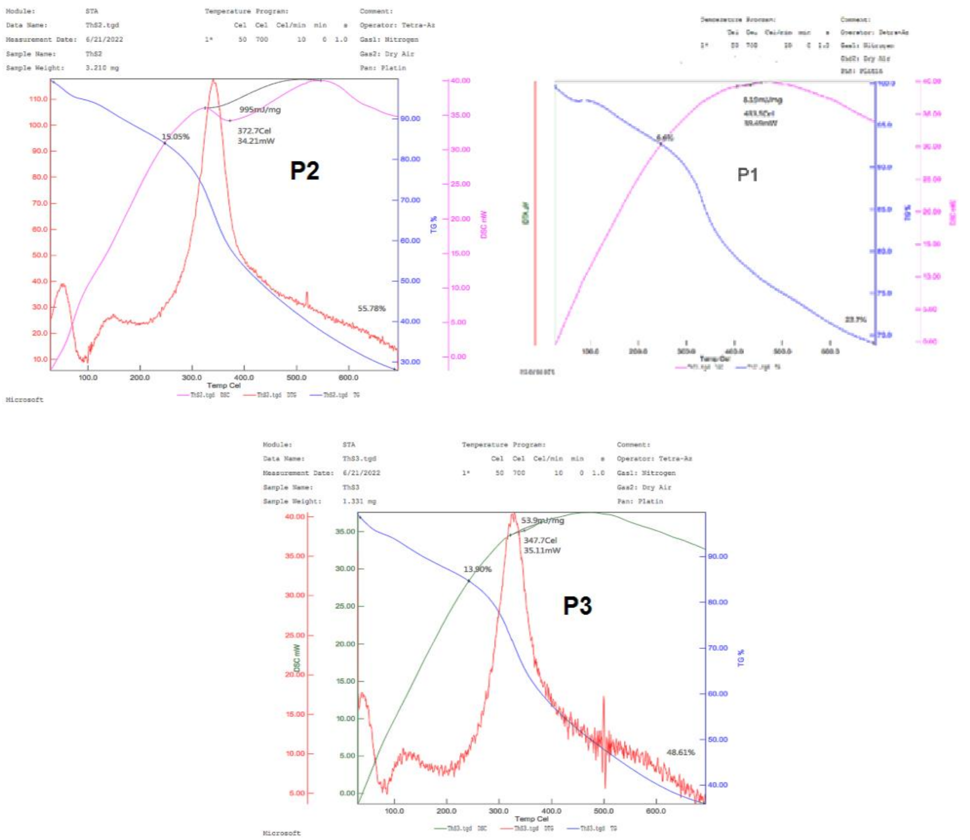
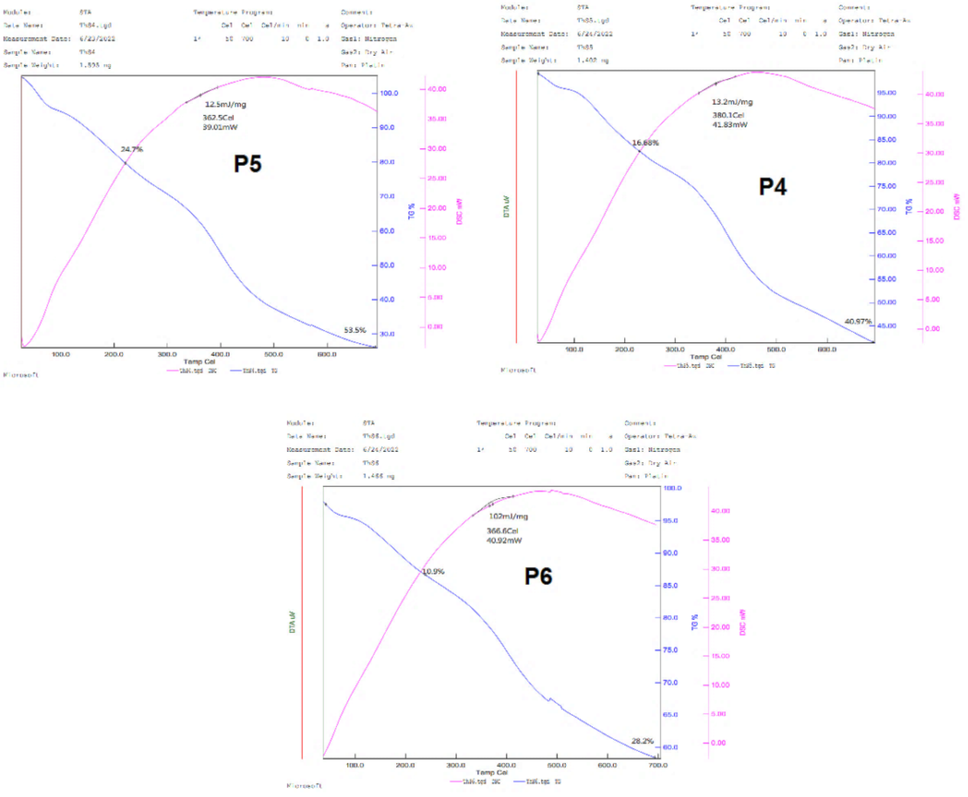


Figure (15): Thermogram of oxadiazole polymers



Figure(16): Thermogram of triazole polymers