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Effect of Azo and Azomethine Moieties on the Electrical Conductivity of Polyesters and Polyamides with Heterocyclic Thiadiazol Polymers

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Abstract:
Five types of polymers containing thiadiazol moiety within their backbone were prepared. Azomethine and azo groups are inserted in the backbone of the polymers. The chemical structures of polymers were studied by using FTIR, also the thermal analysis are measured by TGA method. The electrical conductivity of the prepared polymers were studied. It was shown that the chemical structure of the polymers play the major role affected on the electrical conductivity. The results showed that azomethine and azo groups enhanced the electrical conductivity many times. On the other hand the azomethine moiety increase the electrical conductivity more than that of azo. The results reveals that the amide linking group can enhanced the electrical conductivity more than the ester linking group. Doping the synthesized polymers with two type of dopant (NaI and I_2) was accomplished with different ratios. The electrical conductivity of the doped polymers were increased significantly.
Key words: *thiadiazol polymers, electrical conductivity, azomethine, azo.*

INTRODUCTION:

Heterocyclic Organic conductive polymers (HOCPs), have been the subject of intense investigation due to their applications such as electro-active devices, rechargeable (electrodes) batteries, photovoltaic layers for solar cells, sensors, electrochromic devices anti-static coatings, semiconductors, electrochromic devices and display devices. (1).

HOCPs, In contrast to hydrocarbons, heteroatom containing polymers present additional polarizable donor (O, N), acceptor (S) or ion-conducting properties. Conductive polymers have become a new exciting group of materials discovered due to their interesting properties flexibility in tailoring molecular structures, good solubility in organic solvents, and relatively low cost. and applications. Their sub-unit structure consists of an unfilled π orbital, forming extended π-conjugated systems with low band gaps normally less than 3 eV. Conductivity enhancement is possible by doping with protonic acids, oxidation with metal oxides and reducing agents. (2).

The studies on electrical conductivity of conjugated polymers have been developed very rapidly since the discovery of intrinsically conducting polymers.(3) The conjugation of the polymer chain by Π electrons gives characteristic for the polymer to be oxidized or reduced chemically or electrically [4].

Many generation of conductive polymers have been developed after the discovering of electrical conductivity of polyacetylene (3)as polythiophene and poly(p-phenylene vinylene) (PPV)(5)(6) (7)

Thiadiazoles moiety within the main chain of the polymer offer thermal stability and can influence electronic properties, whereby the sulfur atom can participate in pi-conjugation and can also be involved in redox reactions . [5]

Study shows that the presence of azo moiety within the main chain increase the electrical conductivity of polymers containing some heterocyclic rings.(8). On the other hand increase the number of the azo groups in the main chain enhanced the conjugation, decrease the energy gap and increasing the electrical conductivity(9). The same word can be seen in the presence of azomethine moiety within the main chain (10).

In the present work, polyamide and polyester polymers with thiadiazol moiety within the main chain were prepared. The effect of azo and azomethine groups on their electrical conductivity was studied. The pure polymers was doped with iodine and sodium iodide with different ratios and their electrical conductivity was followed.

Experimental:

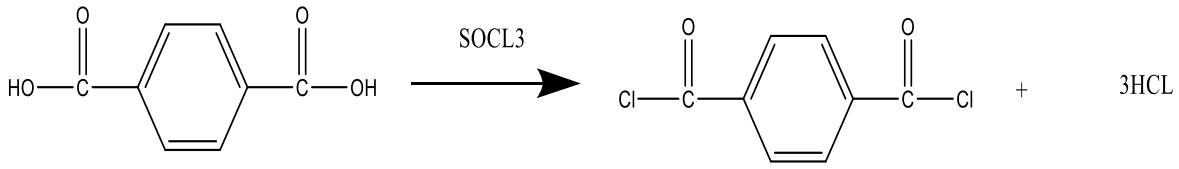
MATERIALS:

All the used chemicals are used as received from the sources without any purification. Table (1)show the used chemicals.

Table 1: Used chemicals

	Source	Materials
1	BDH	Hydrazine Hydrate(80%)
2	BDH	Terephaloyl Chloride
3	BDH	Terephthalic acid
4	BDH	Ammonium Thiocyanate
5	Fluka	Potassium hydroxide
6	Sigma Aldrich	Hydrochloric Acid
7	Fluka	Con. Sulfuric Acid
8	Fluka	Ethanol Absolute
9	Fluka	p-HydroxyBenzaldehyde
10	BDH	Glacial Acetic Acid
11	Fluka	Aniline
12	Fluka	Sodium Nitrite
13	Fluka	Phenol
14	BDH	Acetic Acid
15	BDH	Propionic Acid
16	Sigma Aldrich	Ethyl acetate
17	BDH	Iodine
18	BDH	Sodium Iodide
19	Sigma Aldrich	Hexane

Terephaloyl chloride was prepared by refluxing (0.04 mol) of terephthalic acid with (25) ml of thionyl chloride in the presence of drops of DMF. Refluxing was continued until clear solution was obtained. The excess thionyl chloride was then distilled off and precipitated was recrystallized from normal hexane. The compound was obtained as a white precipitate. M.P (81-82) °C.(11)



METHODOLOGY:

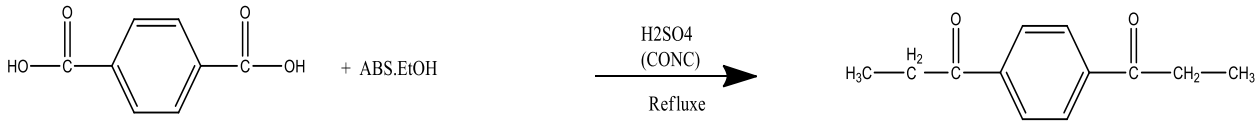
Monomers preparation

Five types of monomers have been prepared starting from monomer (1) 5, 5'-(1, 4-Phenylene) Bis (1, 3, 4-Thiadiazol-2-Amine) , which was prepared from terephthalic acid by series of reaction steps as follows:

Preparation of diethyl terephthalate: (Ⅰ)

The formation of ester of terephthalic acid in absolute ethanol according to published work (12) as followed:

A mixture of (0.2) mole (33.23g) of terephthalic acid dissolved in (250 ml) of absolute ethanol and(16 ml) of conc. sulfuric acid was refluxed for (10-12) hours. The excess ethanol was distilled off, crushed ice (100 ml) was added to the mixture with stirring, the precipitate was filtered, dried, and recrystallized from ethanol. The measured melting point of the dried white product is (58-60) °C. .

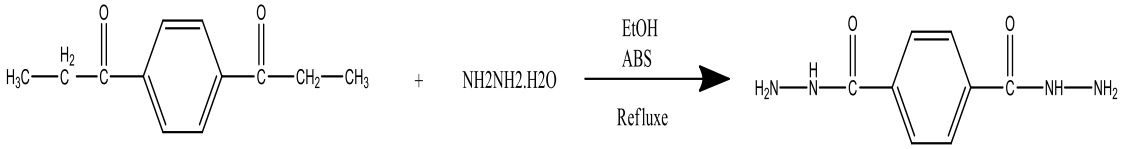


(I)

Preparation of terephthalohydrazide:(Ⅱ)

The formed ester in the last step was reacted with the hydrazine hydrate according to the following procedure: A mixture of (0.04)mole, (8.8896 g) of ester (I), and (60ml) of (0.16) mole of hydrazine hydrate was refluxed for three hours, then 100ml of absolute ethanol was added

and refluxed with the reaction mixture for another three hours. The excess ethanol was distilled off, the precipitate was filtered, dried under vacum and recrystallized form ethanol. The measured melting point of the dried white product was 310 to 312° C [12-15].

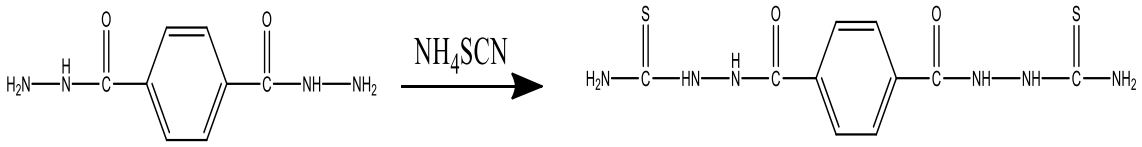


(II)

Preparing the monomer (1):

The method include two steps where by the first step for the synthesis of bis-terephthaloyl thiosemicarbazides (2,2'-terephthaloylbis(hydrazine-1-carbothioamide)) (III) as followes:

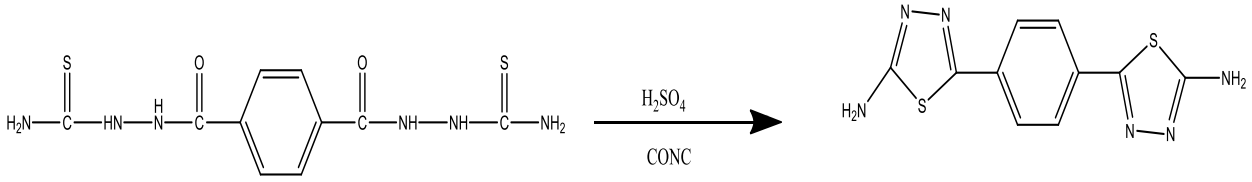
A mixture of (II) (0.01 mol), ammonium thiocyanate (0.02 mol), and (2ml) of concentrated hydrochloric acid in (50mL) absolute ethanol refluxed for 7h., cooling the reaction mixture. The products was filtered and recrystallized from DMF-H_2O (2:1). The white precipitate was dried under vacuum has M.P (188-190°c)(16-25)



III

The second step include the preparation of monomer (1) from(III) as follows:

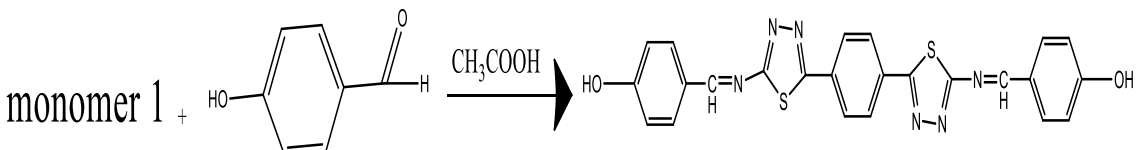
A solution of (III) (0.003 mol) in concentrated sulfuric acid (3 mL) was stirred at room temperature for 2 h and then poured into ice-cold water . The mixture was made alkaline to pH8 with aqueous ammonia and the resultant precipitate was filtered, washed with water and crystallized from DMF-H_2O. The measured melting point of the formed dried precipitate is more than 300°C, its color is white .(26-45).



monomer (1)

Azomethine synthesis (monomer 2) from monomer (1):

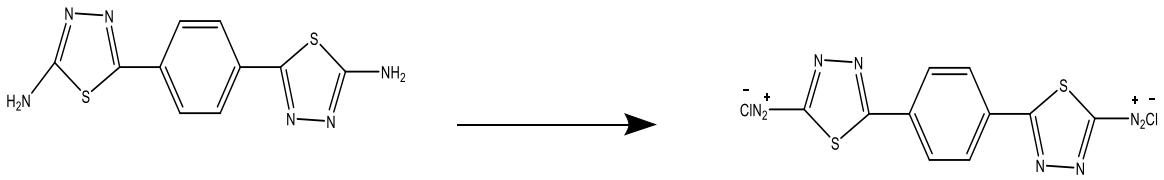
(0.01mole) of monomer (1) was distributed in 35ml absolute ethanol. Thereafter,(0.02mole) of p-hydroxybenzaldehyde was added. six drops of glacial acetic acid into the mixture was added. The mixtures were refluxed for a period of (0.5-1h), the resulted powder was filtered and dried under vacuum. M.P above of 300 ^{0}C**.(12)**



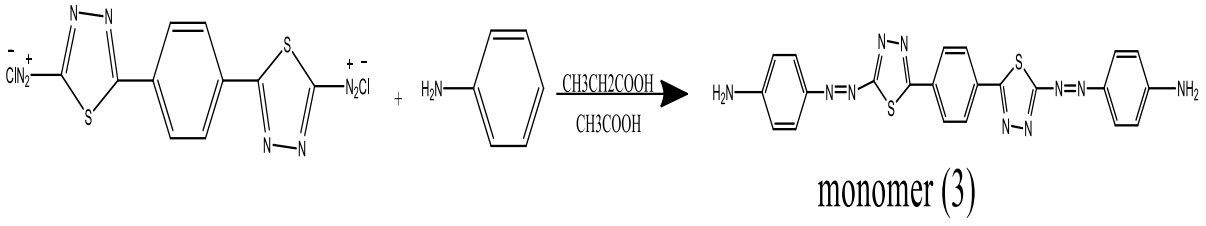
monomer 2

Azotization of monomer (1):

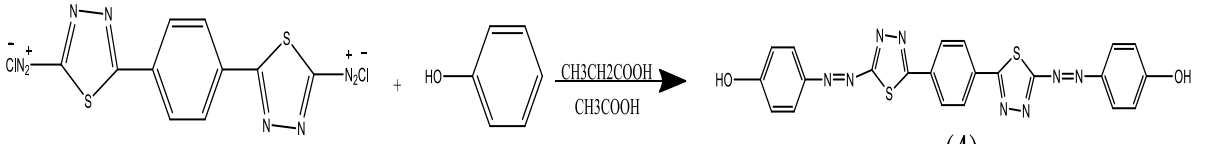
Solution of (0.76 g) sodium nitrite in (5ml) conc. sulfuric acid at 0^{0}C was added drop wise with stirring to (0.01 mole) of monomer (1) to form the diaz onium salt and the temperature was maintained at 0±5 °C for 2 h.



Coupling the Diaz onium salt with aniline and phenol to form monomer (3) and (4) as follows:
To the clear solution of the above Diaz Onium salt was added at 5°C (0.02 mole) of amine solution in acetic acid–propionic acid mixture (4:1, 20 ml), and the reaction was maintained at pH 4 by sodium acetate portion wise addition. Later than a tiny moment, the mixture of reaction was decanted into ice water and filtered. The prepared compounds were washed with water, and dried under vacuum. [47-46]. Also coupling with phenol used same method.

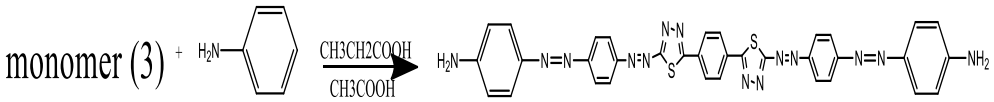


monomer (3)



monomer (4)

Monomer (3) can also be azotized and coupled with aniline by following the same procedure to form monomer (4).

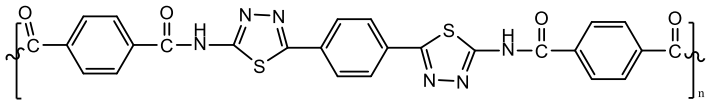


monomer (5)

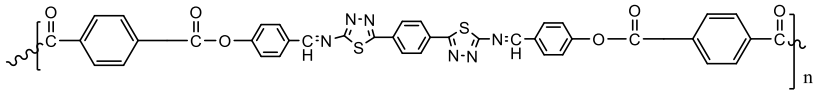
All the prepared monomers have been characterized by FTIR and H-NMR .

Polymerization :

All monomers were polymerized with Terephaloyl chloride by using interfacial polymerization method as follows (49-48) :
(0.01mole) of the monomer was dissolved in aqueous basic solution (10%.). (0.01 mole) of terephtaloyl chloride was dissolved in dry hexane and decant to the monomer solution. The polymer was formed at room temperature, filtered and dried under vacuume.

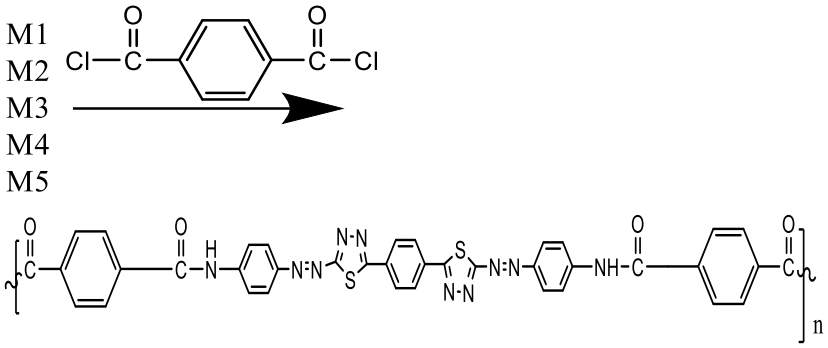


P1

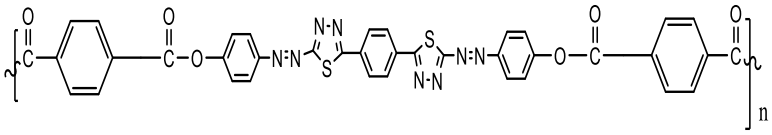


P2

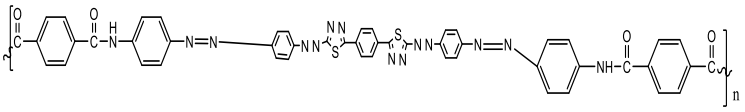
M1
M2
M3
M4
M5



P3



P4



P5

Electrical conductivity measurements:

Films of (2) cm diameter and (0.6-0.8)cm thickness from the synthesized polymers were prepared under (3) ton/cm^2 pressure. The Volume electrical conductivities were measured by using standard three-probe electrical cell method (ASTM35) .

The pure polymers were doped with two different dopant (I_2 and NaI) in order to get N- and P- type conductive polymers respectively:

The doping with NaI with different concentration was done by mixing process , where by the doping with I_2 was followed by using vapor –phase doping . The electrical conductivity of the doped polymers were also measured by the same method.(50).

RESULT AND DISCUSSIONS:

MonomersM1, M2and M3,M4 andM5 have been prepared and characterized by using FT-IR, H-NMR and thermal analysis

Table (2) shows the absorption bands of the special moieties appeared in the IR spectra figures (1-5).

Table(2): FT- IR absorption bands of The prepared monomers:

M	VO-H	NH2	VC=N thiodaizol	VN=N	VC-S	VC-H Ar.	VN-N	VC=N azomethin	VC-N	VC=C
M1	------	3390	1604	------	724	3068	1016	----------	1293	1484
M2	3102	-------	1572	------	686	3025	977	1666	1273	1480
M3	-------	3356	1537	1599	704	3101	989	-----	1300	1503
M4	3334.92	-----	1558.48	1622.13	686.66	3100	990	--------	1392.61	1440.83
M5	-------	3357	1571	1650	715	3057	1001	------	1303	1515

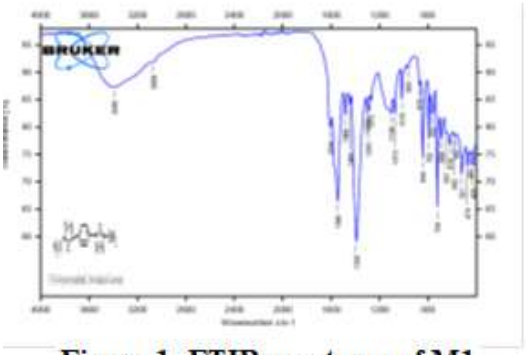


Figure 1: FTIR spectrum of M1

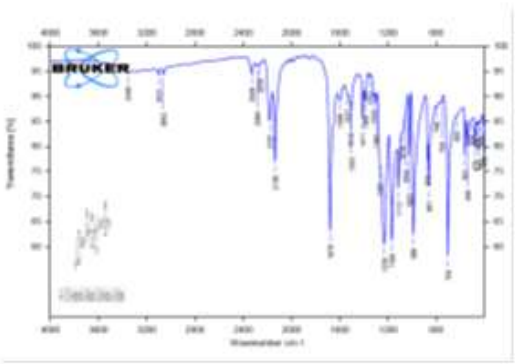


Figure2: FTIR spectrum for M2

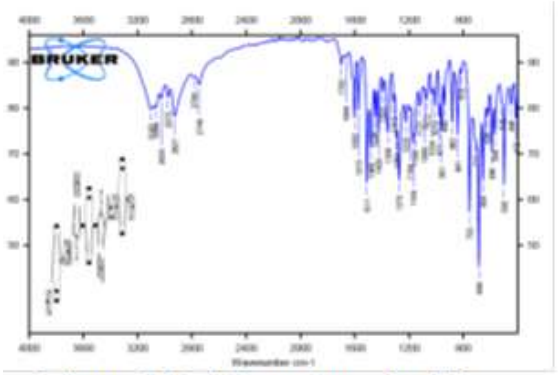


Figure 3: FTIR spectrum for M3

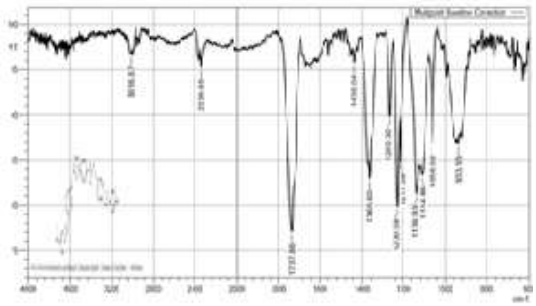


Figure4: FTIR spectrum for M4

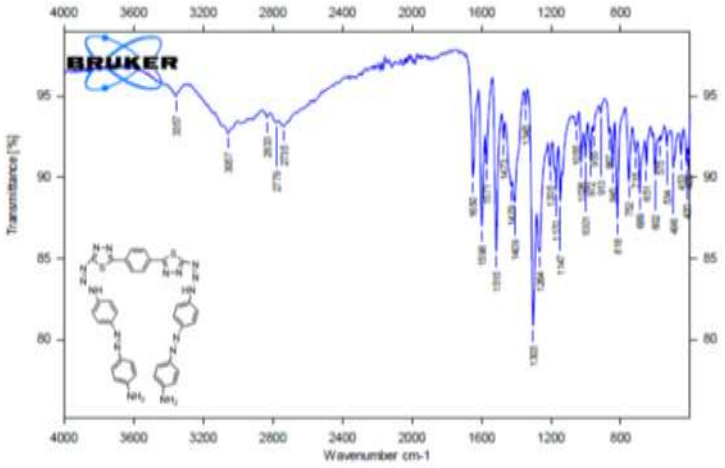


Figure 5: FTIR spectrum for M5

On the other hand, the FT-IR spectrum of the prepared polymers shows a characterized absorption bands as explained in table (3):

Table(3):FT-IR absorption bands of the prepared polymers:

Polymers	VO-H	NH_2	VC=N Thiadiazole	VN=N	VC-S	VC-H Ar.	VN-N	VC=N azomethine	VC-N	VC=C Ar.	VC-N amide	VC=O است	VC-O cm^{-1} استر	VC=O CM-1 کاربونیل امايد

P1	----	------	1504	----	726	3061	1016	----	1386	1504	3419	------	1276	1716
P2	----	------	1412	----	720	3241	909	1599	1306	1486	3241	1682	1306	
P3	----	------	1412	1601	717	2929	910	-----	1305	1516	3017	-------	-----	1684
P4	----	------	1365.60	1590	725	3016.67	1058.3	------	1265.3	1435.0	-------	1737.8	1217.08	------
P5	----	------	1411	1501	701	3022	1023	-------	1305	----	2982	-------	-----	1682

The results revealed that the absorption bands of (NH_2) in monomers (M1 , M3 and M5) (V3390Cm^{-1}, V3356Cm^{-1} and V3357Cm^{-1}) disappeared in the resulted polymers (P1,P3 and P5). In other hands the absorption bands of (OH) in monomers (M2 and M4) (3102Cm-1 and 3334.92Cm-1) also disappeared in resulted polymers(P2and P4).

Figure 6: FTIR spectrum for P1

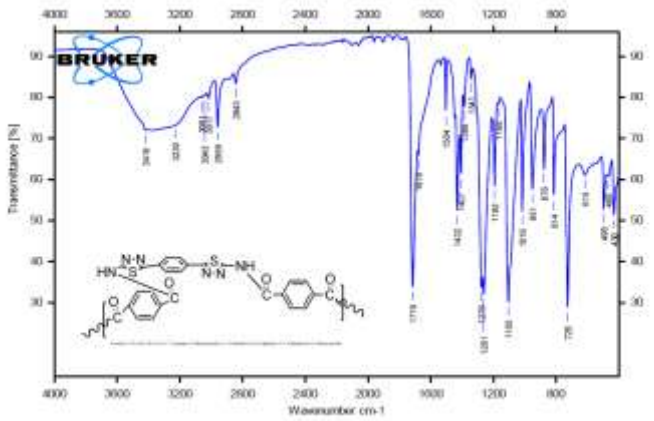
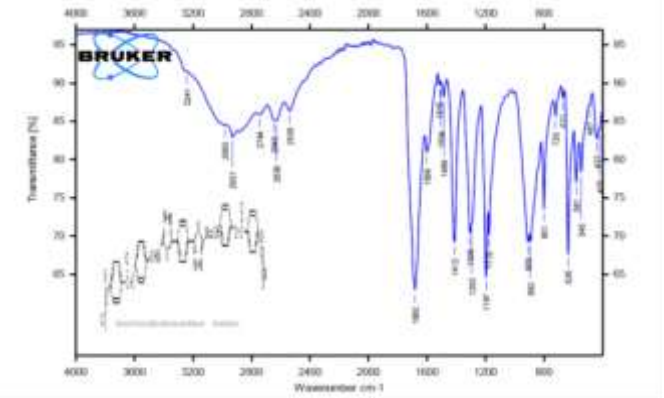


Figure-7: FTIR spectrum for P2



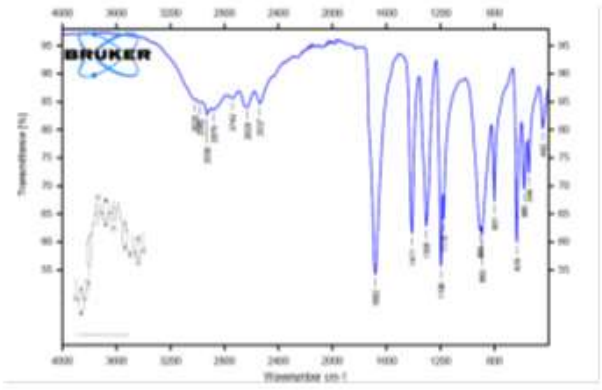


Figure 8: FTIR spectrum for P3

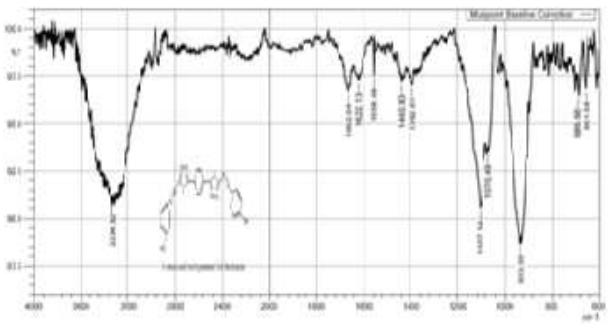


Figure 9: FTIR spectrum for P4

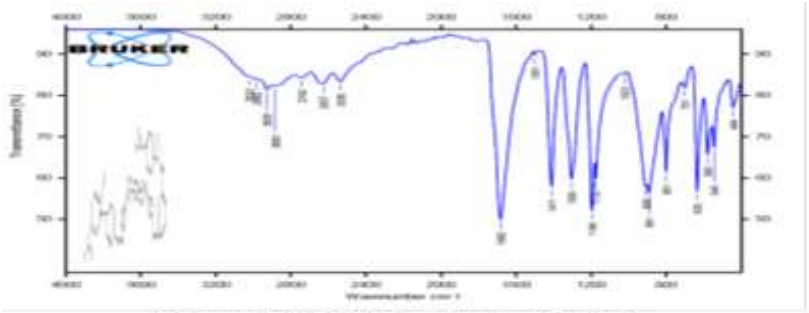


Figure 10: FTIR spectrum for P5

Thermal Analysis:

Three polymers were chosen to study their thermal history by using TGA technique (P1, P2 and P3). Tabl4 (4) and figure (11) shows the thermal stability of the pure polymers. The results reveal that all the polymers have the approximately same behavioure aginst temperature elevation, whereby they can be decompose above 900^{0}C,

Table (4):TGA analysis of the polymers

Polymers	Weight lose %	Temp.\°C	Decomposition\$^0$C
P1	14.3	32.83	905.0
	19.7	356.82	
	21.6	683.8	
	10.6	797.99	
P2	1.2	32.2°C	907.0
	18.51	133.2	
	4.1	250	
	8.8	832.42	
P3	1.3%	40.13C	908
	18.6%	141.2°C	
	9%	394.5°C	
	19.9%	702.5°C	

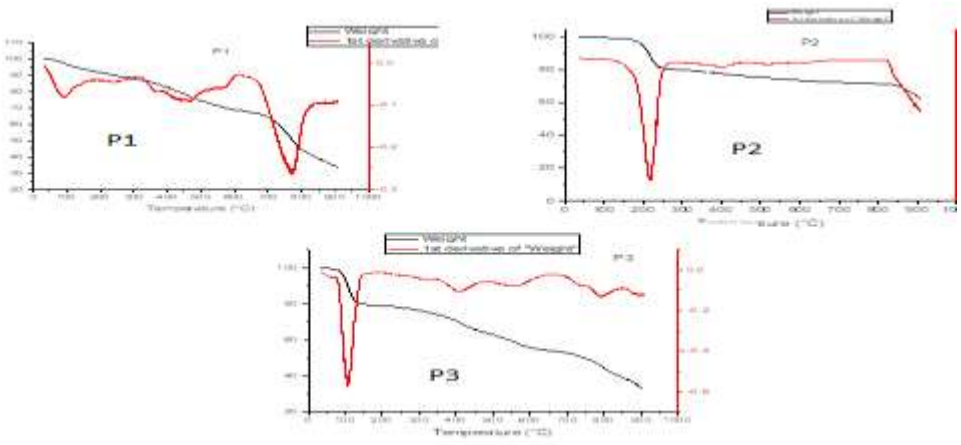


Figure (11): TGA diagram

Electrical conductivity measurement

The Volume electrical conductivity (σ) for the pure and doped polymers were calculating by measuring the current flow in the volume of the specimen at different applied voltage according to the equation: (14)

$\sigma = Id/AE$

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)
E=applied voltage (volt)

Room temperature electrical conductivity of the prepared polymers were shown in the table (4).

Table (4): Room temperature electrical conductivity σ of pure polymer (s/cm)

The results showed clearly that the chemical structures of the investigated polymers can be considered as the controlling factor affected on the electrical conductivity. The electrical conductivity have the order:

polymer	P1	P2	P3	P4	P5
σ x10^{-9}	1.8	44.01	13.5	19.71	21.91

P2 > P5 > P4 > P3 >P1

If we consider P1 is the main polymer, its conductivity was increased when azomethine moiety was present in the main chain (P2), whereby azomethine moiety increase the conjugation of the chain. The conductivity of P2 was decreased when the azo moiety was repleaced the azomethine moiety (P5). This can be explained by that the azomethine moiety enhance conjugation higher than that of azo. On the other hand the type of linking group of the polymers also have a significant effect on electrical conductivity, whereby P5 is polyamide while P4 and P3 are polyester. The most acceptable interpretation is the lone pair of electron in the amide group (P5) can participate in conjugation more than that of ester group(P3 and P4).

Doping effect:

The synthesized polymers were doped with (I_2 and NaI) in order to produce n-type and p-type semiconductor polymer respectively . Tables (5 and 6) show the electrical conductivity variation with different ratios of the dopants.

Table(5): Room temperature electrical conductivity σ (s\cm ×10^{-9}) of polymers doped with different ratio of iodine .(n-type)

polymer	%	σ	%	σ	%	σ	%	σ	%	σ
P1	0.0	1.8	0.141	2.8	0.4	3.7	0.68	4.53	0.861	1.98
P2	0.0	44.01	0.81	67.5	3.9	98.8	6.8	2.3	8.2	0.6
P3	0.0	13.5	1.5	20.21	3.1	85.3	10.0	61.8	49.1	32.4
P4	0.0	19.71	1.8	32.82	29.1	31.96	40.83	31.33		
P5	0.0	21.91	1.03	33.97	3.69	35.98	7.7	26.9		

Table(6):Room temperature electricalConductivity σ(s\cm×10^{-9}) of polymers doped with different ratio of sodium iodide.(p-type)

polymer	%	σ	%	σ	%	σ	%	σ	%	σ	%	σ
P1	0.0	1.8	3.63	21.83	6.99	46.6	10.811	26.14				
P2	0.0	44.01	1.34	60.55	2.48	51.5	3.60	32.22	4.802	20.02	7.52	12.4
P3	0.0	13.5	1.23	98.8	2.95	58.5	4.7	12.61	5.8	4.8		
P4	0.0	19.71	2.05	21.1	4.24	14.3						
P5	0.0	21.91	2.42	32.53	4.7	27.3	7.1	16.3	9.34	9.93		

It was known that the electrical conductivity of doped polymers depends on charge-carrier density and mobility, thus heavily doping a semiconducting polymer possessing high mobility using powerful doping agents is a strategic approach (Kurosawa et al., 20. It is noted from the tables (5 and 6) that the chemical structure of the polymer plays a major role in influencing the electrical conductivity value. The presence of the azo group increases the length of the chain, thus increasing the electrical conductivity. This has been proven by comparing polyamide with polyester- polymers were included or not included azo-factional groups that doped with I_2, P5 with P3, P4 and P 1. We notice that P5>P3>P4>P1This is explained by the fact that the included azo group can increase the polarization value of the chain, thus increasing the electrical conductivity value (Khattab, 2013). However, the conductivity increases even more with the presence of the azo group attached to the aromatic ring Comparison with the absence of azo group. 21)..

(Abu et al.2025)(Ali et al.2025)(Emanuele & D'Auria, 2024)(Modh, 2023)(Elbadawy et al.2025)(Hassan et al., 2024)(Malik & Singh, 2023)(Yang et al.2025)(Rozman & Lukšič, 2025)(Ghamarpoor et al., 2024). In addition, when comparing polyamides and polyesters, we note that the electrical conductivity in polyamides is higher than that in polyesters, as shown P5>P3>P4 .

In addition, when comparing polyamide containing azo groups with polyester containing azomethine groups, we notice that the electrical conductivity of polyamide is greater than that of polyester, as shown P3>P2.

When comparing the polyester polymers, it was observed that the polymer containing the azomethine group has a higher (**σ**) than the polymer containing the azo group,P2>P4 When doping with I_2, the same applies to doping with NaI; the increase in (**σ**) was as follows P2>P4.

When doping with sodium iodide for the amide and ester polymers that contain azo groups, it was found that the (**σ**) increases in the following order,P3>P1>P5>P4 respectively.

CONCLUSION:

Azomethine and azo moieties were introduced into a heterocyclic (thiadiazol) polymer to investigate their effects on electrical conductivity. Analysis of the electrical conductivity measurements indicated that the azomethine component had a larger effect on electrical conductivity. Electrical conductivity can be increased by a doping processes. Doping with iodine and sodium iodide are the most successful procedure.

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