

# Studies on storing and Releasing the Optical Energy via Azo Polymers and its Relation with the Electrical Conductivity

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## ABSTRACT

Nine Azo polymers of different Azo contents and different intra – spacing (aliphatic and aromatic amides or azomethines) were prepared by using interfacial and solution polymerization process. The synthesized polymers were characterized by IR, <sup>1</sup>HMMR, <sup>13</sup>CNMR and thermal analysis techniques. The electrical conductivity and their activation energy were measured by using three probe cell. The optical absorbance measurement were carried out on the thin films of the prepared polymers through irradiation the films by a 20 mw/cm<sup>2</sup> laser beam for appropriate time until saturation take place. It was shown that there is many factors that effects the energy store and release like chain flexibility and number of Azo moieties within the repeating unit. The results showed that there is an inverse relationship between the electrical conductivity and the efficiency of optical storage.

**Keywords:** Azo polymers, optical storage, electrical conductivity.

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## INTRODUCTION

Azobenzene containing polymers received more attentions in last decades for their potential application in many fields, such as optical data storage<sup>[1,2]</sup>, nonlinear optical materials<sup>[3,4]</sup>, holographic memories<sup>[5,6]</sup>, chiroptical switches<sup>[7,8]</sup> and surface relief gratings (SRG)<sup>[9,10]</sup>. The unique commonality among the azobenzene molecules (azos) is the clean and efficient photochemical isomerization that can occur about the azo linkage when the chromophore absorbs a photon<sup>[11]</sup>, the mechanism of this isomerization has been under investigation since early 1950<sup>[12]</sup>. It was first suggested that the azo aromatic groups isomerized by rotation about the –N=N- bond through a  $\pi$  bond rupture. An alternative mechanism was later proposed in which isomerization occurred via the inversion of one or both nitrogen through a linear hybridization transition state in which the double bond was retain. It was generally accepted that the inversion mechanism is involved in the thermal isomerization of azo aromatic groups and both mechanisms are possible in the photochemical isomerization process<sup>[13,17]</sup>.

When azobenzene bounded or doped into polymer matrix, a constant rapid trans –cis-trans photoisomerization activity results in a series of motions, these motions could be classified into three types: molecular, nanoscale and micrometer level. The motion at molecular level is the chromophore one, that results from the interaction between mesogenic Azo molecules and liner polarized light. The second type of motion, at nonscale level, is the motion of chromophore in an organized environment such as liquid crystal or crystalline domains, Langmuir-Blodgett films and nonmolecular films (monolayers). The third type of motion could be obtain by exposing an azo polymer film to a periodic holographic pattern of linearly or circularly polarized light. It was found that massive movement of the polymer chain over long distances (microns) accompanies the photoisomerization of azobenzene<sup>[18]</sup>. Furthermore, a large – scale mass transport of azopolymer chains will occur, which results from the photo induced trans-cis-trans isomerization cycles of azo chromophores<sup>[19-22]</sup>.

Many factors influence the isomerization process have been studied<sup>[23,24]</sup>. In relation to the chemical structure of azo moieties, the bulkiness of the azo groups hinder the chromophore motion and led to a slower writing rate due to the steric effect. Moreover, it was noticed that the influence of azo dipole direction does not play an important role in the isomerization process<sup>[25-26]</sup>. The azo group in the main chain have fewer degree of freedom and exhibit less mobility compared to the azo group in the side chain. The flexibility of the main chain plays a vital role in the photo induced process. In polyesters with sulfone azobenzene, there are similar kinetics of the photo induced with the polymers having an azo moieties in the side chain<sup>[27,28]</sup>.

In the present work, we will study the factors that influence the photo isomerization process, like amount of azo group in the main chain and the types of linkage between them (aromatic and aliphatic amide or azomethine).

## EXPERIMENTAL

### MATERIALS:

Aniline (Fluka), sodium carbonate (Fluka), concentrated hydrochloric acid (BDH), sodium hydroxide (BDH), p-phenylenediamine dihydrochloride (Fluka), sodium nitrite (BDH), terephthalic acid (Koch), terephthalaldehyde (Fluka), phosphorous penta chloride (Fluka), succinic acid (Koch), glutaraldehyde solution (BDH), are purified in accordance with the normal methods<sup>[29]</sup>. All the solvents are used as received from the commercial sources. Terephthaloylchloride (TDC) is prepared from reaction of terephthalic acid (4.2 g) with phosphorous penta chloride (11g), reflux the mixture for (30 min.), POCl<sub>3</sub> has been separated by distillation at 115°C, the remaining product (TDC) is purified by recrystallization from normal hexan, the melting point is 81°C. Succinyl chloride is prepared by reaction of (8.34 g) phosphorous penta chloride with (2.36 g) of succinic acid, POCl<sub>3</sub> is separated by distillation. The precipitated (succinyl chloride) is filtrated and washed with benzene. TGA and DSC measurements were carried out by using extra TG/DTA 6300 apparatus

### SYNTHETIC ROUTE

#### TETRAZONIUM MONOMERS PREPARATION:

Monomer (I) was prepared as follows :

0.36 g ( $1.773 \times 10^{-3}$  mol) of p-phenylenediamine dihydrochloride was dissolved in (2 ml) of water and (20 ml) of conc.HCl, an aqueous solution of (0.30 g) sodium nitrite was added drop wise during 30 min. with stirring at -10°C. ,the stirring was continued for 30 min. at this temperature to form a suspended solution. (4ml) of pure aniline was added to the suspended solution and stirred for 1.5 hr., the precipitated monomer (I) was filtered, washed with ether and dried under vacuum. To prepare monomer (II), monomer (I) was used instead of p-phenylenediamine dihydrochloride by following the same procedure. Monomers (III-IV) were prepared from monomers (II-III) respectively by using the same method.

#### POLYMERS PREPARATION:

##### POLYAMIDES:

Polymer (I) was prepared as follows: Dissolving 0.20 g ( $0.514 \times 10^{-3}$  mol) of tetrazonium monomer (I) in 5 ml of 2% sodium hydroxide solution under cooling. Another solution of 0.12g ( $0.514 \times 10^{-3}$  mol) of (TDC) dissolved in 5 ml benzene was added to the first solution under stirring for 30 min. at room temperature. The obtained precipitated polymer was filtrated and dried under vacuum. Polymers (II-IV) were obtained from tetrazonium monomers (II, III, IV) respectively by using the same procedure. Polymer (VIII) was prepared by polymerizing tetrazonium monomer (I) with equimolar of succinyl chloride by following the same method.

##### AZOMETHINE POLYMERS:

Polymer (V) was synthesized by dissolving 0.76 g ( $1.979 \times 10^{-3}$  mol) of tetrazonium monomers (I) in ethanol, bubbling nitrogen gas for 5 min., 0.26 g ( $1.979 \times 10^{-3}$  mol) of terephthalaldehyde was added to the solution and refluxed for 30 min. Benzene was added drop wise to the reaction mixture and distilled off at the same rate of adding. The precipitated polymers was filtered and dried under vacuum. Polymers (VI), (VII) have been prepared by the same method from tetrazonium monomers (II) and (III) respectively, while polymer IX was prepared by the reaction of tetrazonium monomer I with glutar aldehyde by the same method. All the prepared monomers and polymers have been characterized by using F.T.IR spectrophotometer, (biotech engineering management CO.LTD., UK), <sup>1</sup>HNMR and C<sup>13</sup> NMR (Brucker ,300 MHZ).

### MEASUREMENT:

#### THE ELECTRICAL CONDUCTIVITY MEASUREMENTS:

Films with 2 cm diameter and 0.5 mm thickness from the synthesized polymers are prepared under of 4-5 ton / cm<sup>2</sup>. Electrical volume conductivity measurements are performed, using the standard 3-probe DC. technique according to the ASTM method<sup>[30]</sup>.

### THE OPTICAL MEASUREMENTS:

A thin films of the synthesized polymers have been accomplished on a precleaned glass substrate using spin coating technique from 10% DMF solution at 4000 r/min. rotation. The specimens were kept in dark and dried atmosphere until conducting the optical measurements. The specimens are irradiated with a laser beam of 20 mW/cm<sup>2</sup>. The maximum absorption was measured during irradiation until saturation. The laser then was turned off and the absorption was followed until a constant absorption was reached.

## RESULTS AND DISCUSSION

### MONOMERS SYNTHESIS AND CHARACTERIZATION:

Scheme 1 illustrates the synthetic route of the tetrazonium monomers. Tetrazonium monomer (I) was prepared from p-phenylenediamine dihydrochloride. The synthesized monomers were characterized by IR spectroscopy. The IR spectrums of the monomers show a peak at about (1400±10) cm<sup>-1</sup> and at (3250-3550) cm<sup>-1</sup> which are related to the azo and NH<sub>3</sub><sup>+</sup>Cl<sup>-</sup> groups respectively for all monomers. The peak appeared at 1250 cm<sup>-1</sup> is belong to the C-N group, while the C=C aromatic are characterized by four bands appeared between (1459-1600) cm<sup>-1</sup>.

Table (1) shows the chemical structures of the prepared polymers. Interfacial polymerization has been used to prepare the polymers (I, II, III, IV) while the other polymers have been prepared by solution polymerization process. Polymers (I, II, III, IV) are azo polyamides prepared from the counterparts tetrazonium monomers respectively with terephthaloyl chloride while polymers (V, VI, VII) are aromatic polyazomethine prepared from tetrazonium monomers (I, II, II) respectively with terephthalaldehyde. Polymer (VIII) is polyamide with aliphatic segment prepared from monomer (I) with succinyl chloride while polymer (IX) is polyazomethine with aliphatic group prepared from tetrazonium monomer (I) with glutaraldehyde solution. The IR spectrum of the polymers show a new characteristic bands which are belong to the functional groups formed through polymerization process. Table (2) shows the frequency of the different functional groups appeared via IR spectrum. Table (3) and Table (4) indicates the frequency appeared at the <sup>1</sup>HNMR and <sup>13</sup>CNMR spectrum of the investigated polymers.

### THE ELECTRICAL CONDUCTIVITY MEASUREMENTS:

#### CHEMICAL STRUCTURE EFFECT:

Table (5) shows the ohmic electrical conductivity of the prepared polymers at (1000 V) external applied field. The result indicates that the chemical structure of the investigated polymers play an important role effecting the measured conductivity. Increasing the azo moieties content within the backbone of the polymers chain led to increase the polarization of the chain and enhancing the conductivity. This can be shown clearly on going from polymer I to II. It was also monitored that polymer (IV) has a different structure in the linkage group between the azo moieties (azomethine group). The value of its conductivity will be decreased whereby the azomethine group will decrease the chain flexibility and reducing the conductivity<sup>[31]</sup>.

The conductivity values of polymers (V, VI, VII) are decrease with increasing the length of the azo group. This result can be explained by that the azo group could be consider as a rigid rod, increasing its ratio in the main chain will decrease the flexibility and decrease the conductivity. In comparison between polymers I, II and polymers V, VI respectively, it was clearly noticed that the connecting groups between azo moiety have a significant effect on conductivity. The azomethine group in polymers V and VI support the conjugation factor in the chain and enhancing the conductivity more than the amide group in polymer I and II. Inserting aliphatic group within the main chain, as in polymers VIII and IX will increase the flexibility of the polymers chains and increase the conductivity values as in comparison with the related polymers with complete aromatic moieties via the main chain (polymers I and V respectively).

#### CHANGE OF CONDUCTIVITY WITH TEMPERATURE ELEVATION:

Table (6) shows the electrical conductivity of the investigated polymers at different temperatures. There are a systematic increase of electrical conductivity with the temperature, whereby the population of electrons in conduction band increased with temperature elevation<sup>[32]</sup>. The calculated activation energy derived from relation between log σ vs. the inverse absolute temperature was shown in table (7). The increase of conductivity with elevation in temperature could be attributed to two factors. The first one is the rise of population of electrons from HOMO to LUMO levels. This mechanism need activation energy of about (1.5-2)eV<sup>(33)</sup>. The second factor, is the increase of the mobility of chain segments with increase the temperature<sup>(34)</sup>. The activation energy of the second factor is near the determined one in this work.

The values of the activation energy for polymers I to IV is very little (0.060-0.015). Therefore the increasing in the conductivity with temperature elevation could be explained in terms of increasing the segmental mobility with temperature elevation. The results revealed that the azo moieties have two reversible factors affect the alteration of the

electrical conductivity with the temperature elevation. The positive factor is increasing the azo contents increase the length of conjugation of  $\pi$  electrons and support for the diffusion of the electrons from HOMO to LUMO as it was noticed on going from polymers I to II. The other factor which is negative, whereby the azo moieties is a rigid rod and introduce a stiffness in polymer chain, the presence of a large scale of this moiety (polymer IV) led to decrease the flexibility of the polymeric chain and producing a very low value of activation energy or in the other word increase in the sensitivity to temperature<sup>(35)</sup>. The high values of the activation energy of polymers V, VI, VII (0.504, 0.631, 0.630 respectively) could be explained by the fact that the increasing of conductivity with temperature is basically resulting from the increasing the flexibility of chains more than the increasing of the diffusion of the electrons from HOMO to LUMO, since the structure of these polymers contain azomethins groups instead of the less flexible groups (the amide groups)<sup>(36)</sup> The high activation energy value for polymers (VIII) and (IX) could be attributed to the presence of aliphatic group within the main chain which will increase the flexibility and leading to increase the activation energy.

### THERMAL ANALYSIS:

The TG and DTG thermograms of polymers II, VIII and IX are shown in figure (1). Table (8) shows the thermal constants of the polymers calculated from the TG spectrum, where the  $id_t$  represent the starting degradation temperature,  $od_t$  is the maximum degradation temperature and  $fd_t$  is the final degradation temperature. It was clearly observed that the chemical structure of the polymers have a significant effects on their thermal constants.

The  $id_t$  of the polymers have the order:  
II>VIII>IX

The aliphatic segment in polymers VIII and XI lowers their  $id_t$  less than that of polymer II. The presence of amide linkage in polymer VIII introduce a stiffness in polymer chain more than the azomethine moiety via polymer IX The glass transition temperature of the investigated polymers deduced from the DSC curves (figure 2) are shown in table (9). It was clearly observed that polymer I and II have maximum ( $T_g$ ), since aromatic ring and amide group are moieties predominantly reduce the flexibility of the chain. The  $T_g$  of polymer (V) was less than that of polymer (I) and (II), the reasons can be related to the formation of hydrogen bonding by amide group in polymers I and II. The presence of aliphatic group in the main chain as in polymers (VIII), (IX) will increase the flexibility and decreasing the ( $T_g$ ) value.

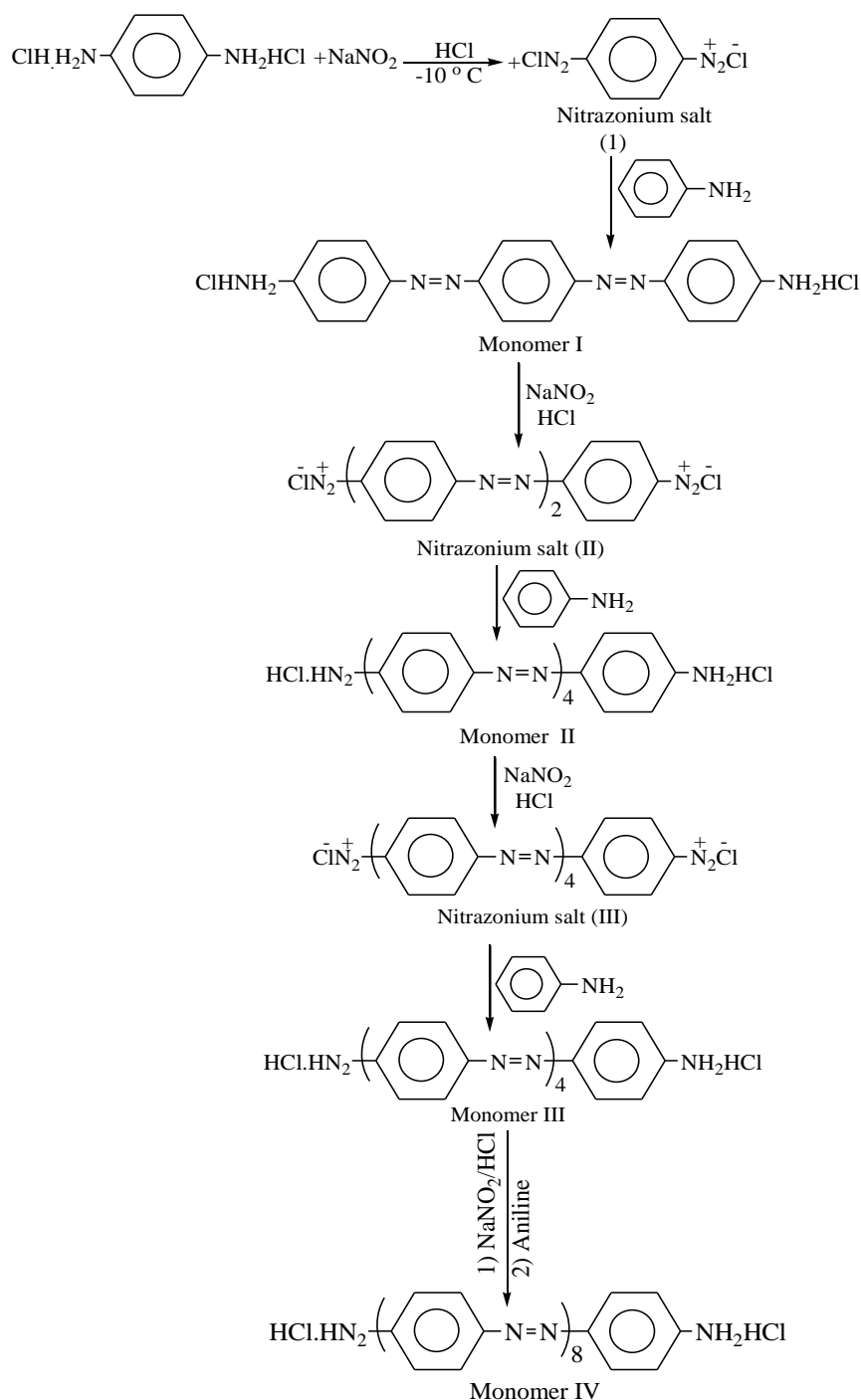
### THE OPTICAL MEASUREMENTS

The cis-trans isomerization within the azo group can be estimated as an optical energy storage process. This behavior of the studied chromophores was investigated at room temperature by means of photolysis. The polymer films were irradiated with a linear polarized beam of a green laser light with  $20 \text{ mW/cm}^2$ . Before irradiation, the azo moieties are exist in the more energetically favored trans form. Since the laser beam is turned on, photoisomerization is induced and photostationary state of both trans and cis isomers is reached. This causes a sharp decrease in the absorbance and hence an increase in the light intensity passing through the sample. Once the beam is turned off, thermal cis-trans isomerization spontaneously follows and puts the system back into the more stable trans isomer<sup>(37)</sup>

Table (10) shows the changes in the absorbance of the polymers films through irradiation until saturation and after the beam was turned off through following the absorbance with the time. We can postulate that the time of irradiation until saturation is the time needed to store the sufficient energy in the sample ( $T_1$ ). The time after the laser is turned off until the absorbance reaches the original absorbance before irradiation can be represent the time needed to lose the energy storage via irradiation ( $T_2$ ) or relaxation time. On the other hand the difference between the absorption before and after the irradiation could be account as the amount of the energy stored by the sample.

The results revealed that the chemical structures of polymers chains play amajor role on the cis-trans isomerization process. Flexible spacers within the polymer chain between the azo group assists to store and release the energy by reducing  $T_1$  and  $T_2$  as shown in comparison between polymer I with polymer VIII or polymer V with IX. This can be explained by that the flexible spacer enhanced the formation of an amorphous phases and producing an anisotropic optical properties. The result shows that increasing the azo moieties within the repeating unit on going from polymer I, II, III, to IV or from polymer V, VI, to VII led to reduce the amount of the stored energy, but there are no systematic effect on  $T_1$  or  $T_2$ . In according to table (10), it has been observed that there are some relations between the electrical conductivity and the studied optical properties. The explanation could be attributed to that the two processes include exposing the sample to external field. The external field induce the formation of a dipole within the azo group and enhancing the electrical conductivity and oriented the dipoles to form an optically active axis. In the other word the factors that enhanced the electrical conductivity makes the isomerization processes easier.

It was observed that on going from polymers V to VI to VII, whereby the azo moieties content increase, there is a direct proportion between electrical conductivity and the amount of the stored energy. This can be explained by that the enhancing of the electrical conductivity will make the driving of cis isomer to trans one more easy.



**Scheme (1): Synthetic route of tetrazonium monomers**


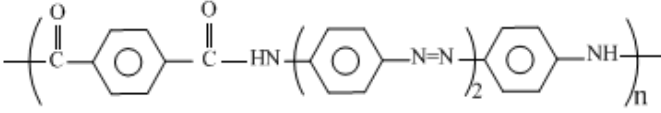

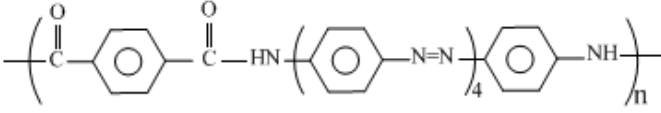

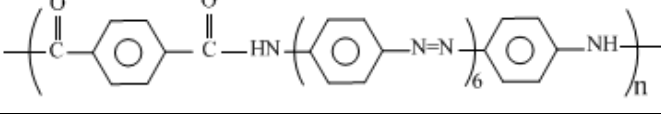
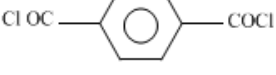
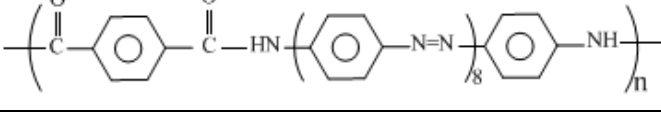

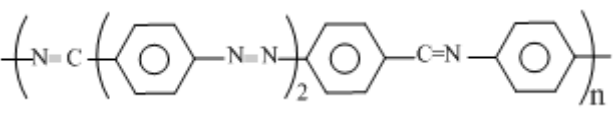
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Table (1): Chemical structure of the prepared monomers and polymers.

No. of polymer	First monomer	Second monomer	Polymer structure
I	I		
II	II		
III	III		
IV	IV		
V	I		

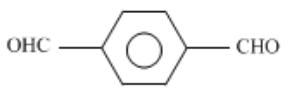
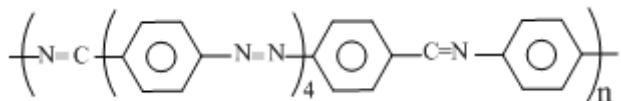
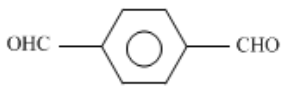
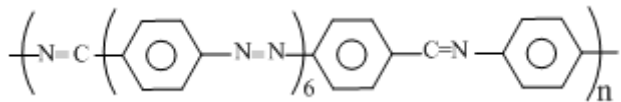
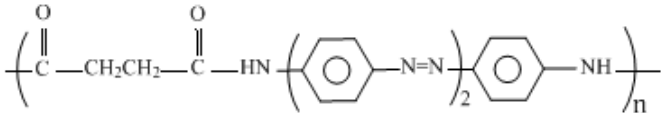
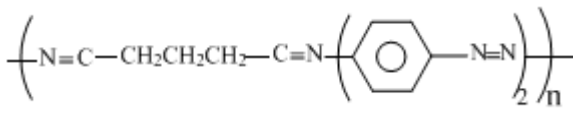
VI	II		
VII	III		
VIII	I	$\text{ClOC}-(\text{CH}_2)_2-\text{COCl}$	
IX	I	$\text{OHC}-\text{CH}_2\text{CH}_2\text{CH}_2-\text{CHO}$	

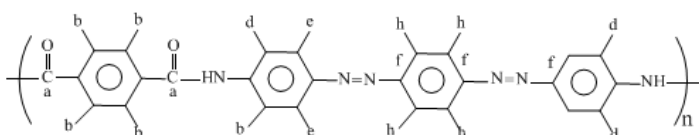
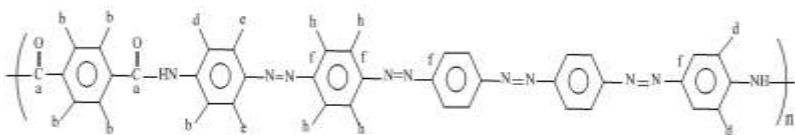
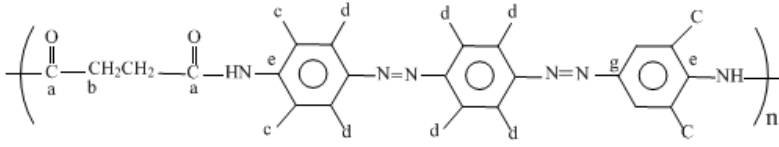
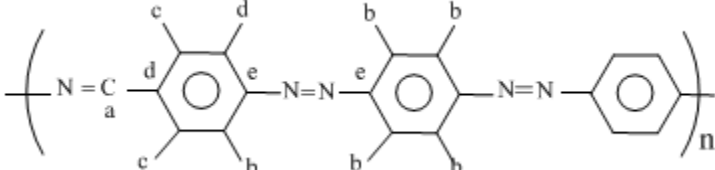
Table (2): F.T.IR band frequencies of the prepared polymers.

Polymer	C=O	Bending NH-	C=C aromatic	N=N	C=N	Str. aliphatic C-C
I	1650	1599	1529	1400	----	----
II	1649	1620	1533,1506	1402	----	----
III	1649	1601	1439,1535,1502	1400	----	----
IV	1647	1600	1500,1533,1580	1441	----	----
V	----	----	1618,1498	1387	1653	----
VI	----	----	1616	1493	1650	----
VII	----	----	1616	1493	1650	1340
VIII	1645	1618	1541,1442	1438	----	----
IX	----	----	1616	1493	1638	1194

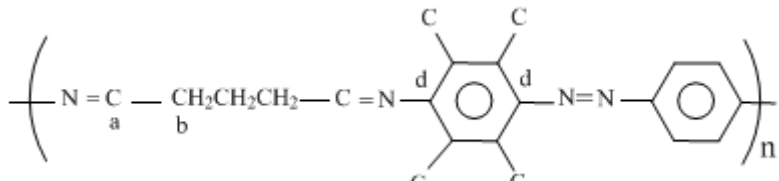
Table (3): HNMR bands of the prepared polymers.

Polymer	(H)aromatic	(-NH)amide	N=CH
I	8.121-6.996 4 Peaks	10.407	----
II	8.115-7.105 4 peaks	10.407	----
V	8.232-6.875 Multiple	----	10.421
VIII	7.702-6.733 Multiple	10.067	----
IX	7.498-7.386	----	10.318

Table (4): <sup>13</sup>CNMR bands of the prepared polymers.

Polymer	Frequency	Structure
I	165.26 (a) 128.18 (b) 139.44 (c) 124.35 (d) 120.94 (e) 137.90 (f)	
II	156.26 (a) 128.18 (b) 139.44 (c) 124.35 (d) 120.94 (e) 137.90 (f)	
VIII	170.85 (a) (b)116.83) (c)79129.) 127.11 (d) (e)139.83) 124.38 (f)	
V	132.4 (a) (b)123.7) 130.2 (c) 128.5 (d)	



<b>IX</b>	133.02 (a)	
	(b)123.35	
	130.11 (d)	
	127.98 (c)	

**Table (5): Electrical conductive of the pure polymers measured at 1000 v .**

Polymer	( $\sigma$ )/Ohm <sup>-1</sup> cm <sup>-1</sup>	Polymer	( $\sigma$ )/Ohm <sup>-1</sup> cm <sup>-1</sup>
(I)	5.868*10 <sup>-12</sup>	(VI)	7.571*10 <sup>-8</sup>
(II)	7.042*10 <sup>-11</sup>	(VII)	3.051*10 <sup>-9</sup>
(IV)	4.884*10 <sup>-11</sup>	(VIII)	5.621*10 <sup>-11</sup>
(V)	3.459*10 <sup>-7</sup>	(IX)	6.270*10 <sup>-10</sup>

**Table (6): Effect of temperature on the electrical conductivity of the polymers at 1000 v.**

Polymer	( $\sigma$ )/10 <sup>-11</sup>	(T)°C	Polymer	( $\sigma$ )/10 <sup>-11</sup>	(T)°C
<b>I</b>	2.084	58.8	<b>VI</b>	1.140	62.5
	2.738	71.4		3.663	76.9
	4.333	90.9		4.316	90.9
				4.316	100
<b>II</b>	0.85	62.5	<b>VII</b>	983.8	47.6
	0.73	83.3		160.3	66.6
	1.501	100		1200.00	83.3
	1.300	105		3593.00	100
<b>IV</b>	0.90	52.6	<b>VIII</b>	4.00	40
	1.022	71.4		26.8	83.3
	1.448	90.9		94.00	100
	1.737	100		820.1	107
<b>V</b>	579.00	50	<b>IX</b>	59.00	83.3
	243.00	66.6		180.00	102
	475.00	90.9			
	227.00	108			

**Table (7): Activation energy for the electrical conductivity.**

Polymer	$\Delta E/eV$	Polymer	$\Delta E/eV$
(I)	0.060	(VI)	0.630
(II)	0.084	(VII)	0.631
(IV)	0.015	(VIII)	0.78
(V)	0.504	(IX)	0.536

**Table (8): Thermal constant of the polymers calculated from TGA thermo gram.**

Polymer	idt /c°	odt /c°	fdt / c°	% Char content
II	375	450	600	14.4%
VIII	212	300	550	23.1%
IX	198	484	550	9.8%

**Table (9): Tg of the polymers.**

Pol	Tg / c°
I	215
II	260
V	186
VIII	118
IX	75

**Table (10): Optical measurement of the polymers**

P0lymer	A1	A2	T1/min.	T2/min.	Energy gained	( $\sigma$ ) / Ohm <sup>-1</sup> cm <sup>-1</sup>
I	2.02	1.5	33	65	0.52	5.868*10 <sup>-12</sup>
II	1.04	0.96	30	66	0.08	7.042*10 <sup>-11</sup>
III	1.12	1.07	38	95	0.05	
IV	1.08	0.93	27	120	0.15	4.884*10 <sup>-11</sup>
V	1.08	0.99	20	34	0.09	3.459*10 <sup>-7</sup>
VI	1.38	1.36	56	60	0.02	7.571*10 <sup>-8</sup>
VII	1.34	1.33	38	68	0.01	3.051*10 <sup>-9</sup>
VIII	0.87	0.83	30	60	0.04	5.621*10 <sup>-11</sup>
IX	0.83	0.69	11	16	0.14	6.270*10 <sup>-10</sup>

A1=absorbance before irradiation, A2= absorbance after irradiation