

**Preparation and Characterization Some Monomers and Polymers Derived from
Azo - Schiff Base Compounds and Studying Liquid Crystalline Properties, Electrical
Conductivity and Dielectric Constant**

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Properties, Electrical Conductivity and Dielectric Constant**

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Abstract

This research included the preparation of some monomers and polymers by using (*P*-phenylene di amine) (A) as central unit (nucleus) for the preparation of these compounds in several steps following, the first step included the preparation of new derivatives of compounds (B) and (C), The second step included the preparation of three series of schiff bases (A₁- A₅), (B₁-B₅) and (C₁-C₅), The third step included polymerization of monomers that prepared in the 2nd step (A₁ , A₂ , B₁ , B₂ , C₁ , C₂), the prepared compounds diagnosed were by spectroscopy methods which includes infrared spectroscopy (IR) and some compound using nuclear magnetic resonance of the proton (¹H-NMR), as well as the liquid crystalline state of the compounds prepared above studied was obtained compounds in (Nematic phase) and observed changes at different temperatures, as well as measure the real dielectric constant of these compounds, and the values obtained are excellent as these compounds have high dielectric making it store materials for energy.

Key words: liquid crystal, Schiff base, Conductivity, Real dielectric constant

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تحضير و تشخيص مونمرات و بوليمرات مشتقة من مركبات الأزو - قواعد شف و دراسة خصائصها البلورية سائلة و التوصيلية الكهربائية و ثابت العزل الحقيقي

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الخلاصة

تضمنت الدراسة تحضير بعض المونمرات والبوليمرات بإستعمال (P-phenylene di amine) المركب (A) مركباً أساسياً (نواة) لتحضير هذه المركبات باتباع عدة مراحل شملت المرحلة الاولى تحضير مشتقات جديدة من مركبات الأزو (B) و (C) ، بينما تشمل المرحلة الثانية تحضير ثلاث سلاسل من قواعد شف (A₁-A₅) و (B₁-B₅) و (C₁-C₅) و شملت المرحلة الثالثة بلورة المونمرات (A₁ , A₂ - B₁ , B₂ - C₁ , C₂) ، تم تشخيص المركبات المحضرة بإستخدام طرق طيفية مثل طيف الأشعة تحت الحمراء (IR) ولبعض المركبات بإستعمال طيف الرنين النووي المغناطيسي للبروتون (¹H-NMR) ، كذلك دراسة الحالة البلورية السائلة للمركبات المحضرة اعلاه إذ تم الحصول على مركبات في الطور النيماتى و لوحظت تغيرات الأطوار للمقاطع النسيجية عند درجات حرارة مختلفة ،أيضاً تمت دراسة التوصيلية وثابت العزل للمركبات المحضرة وعند مدى واسع من التردد إذ وجدت بعض القيم للتوصيلية عند ترددات معينة هي جيدة , وكذلك حسب ثابت العزل الحقيقي لهذه المركبات و كانت القيم التي حُصل عليها ممتازة إذ كان ثابت عزل هذه المركبات عالٍ مما يجعلها مواد خازنة للطاقة.

الكلمات المفتاحية: البلورات السائلة، قواعد شف، التوصيلية، ثابت العزل الحقيقي

Introduction

Liquid crystals are an intermediate state (Mesophase) [1,2], it has been described as the fourth state of matter it was named at First fluid crystals (flowing crystals) this situation occur between the solid phase which the motion of molecules be constrained in the tri- dimensional network and with integrated molecular regulation position and direction, the isotropic phase which the particles move freely and be random organization, the Austrian botanist (Friedrich Reinitzer) (1888) [3], is the first to notice this case when his study of cholesterol benzoate as it was found that this material is fused to mutant turbid liquid at a temperature (146 C) but fully fused at a

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temperature (179C) it turns into liquid so it named isotropic liquid crystals are classified into two categories to (Lyotropic) and (Thermotropic) depending on the method by which gets smashed crystalline grid system [4], the nematic phase derives its name from a Greek word nematos and its meaning (Filar or wired) , the nematic liquid crystals is the simplest liquid crystalline eccentric and is symbolized (N phase) [5], and in which the molecules possess a salary system the molecules in nematic phase have over a large turnover [6,7], the most common application of liquid crystal is liquid crystal displays, which rely on the optical properties of certain liquid crystalline molecules in the presence or absence of an electric field [8], and it used in medical applications show change of color- coordinated with the temperature [9], more important and practical applications have been developed in such diverse areas as electronics [10], and also Liquid crystal in fluid form is used to detect electrically generated hot spots for failure analysis in the semiconductor industry[11], Azo Schiff base conjugated polymers (CPs) are organic macromolecules characterized by a backbone chain consisting of alternating single- and double-bonds. Their overlapping p-orbitals create a delocalization system of π -electrons thus resulting in interesting and useful electrical properties [12], the purpose of the present study is to prepare monomers and polymers that liquid crystalline properties and it have electrical properties (conductively, electrical energy storage) to use in the electronics applications.

Experimental

A materials which are used in chemical reaction characterization by FT-IR, known purity and measured M.P before used it.

Prepare (B, C) compound

Prepare diazonium salt

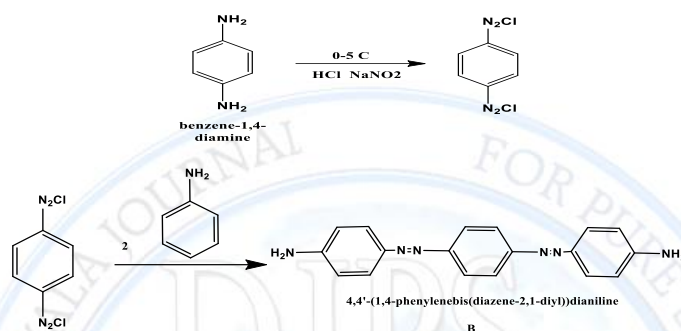
2 ml of (Conc. HCl) with 10 ml of distilled water were dissolved in (0.324 g – 0.0003 mol) of (*P*-phenylene di amine) at (0-5 °C), in the second flask prepared solution by dissolved (0.456 g) of sodium nitrite in 5 ml of distilled water at (0-5°C), then added the first solution to the second solution as batches ,and the temperature at (0-5°C) .

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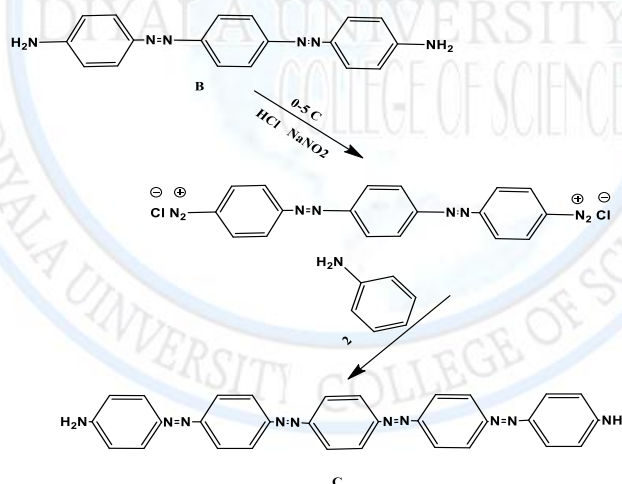
Coupling Reaction

In 5ml of (35% NaOH) dissolved (0.558 g – 0.0006 mol) of aniline at (0-5 °C) and then added to diazonium salt with stirrer at (0-5°C) ,to obtained dark brown precipitate.



Scheme 1: The preparation (B) compound

The (C) compound prepared by used the same method above of prepare (B) compound.



Scheme 2: the preparation (C) compound

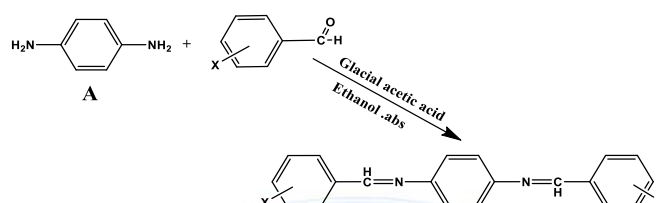
Prepare Schiff base monomers (A₁-A₅)

The synthesis of Schiff base is schematically presented at (schem 3) 10 ml of an ethanolic solution of (*P*-phenylene di amine) (0.324 g - 0.003 mol) was added to 10 ml of an ethanolic solution of (*O*-hydroxy Benzaldehyde) (0.636g – 0.006mol) with few drops of glacial acetic

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acid. the mixture was refluxed for 4hrs.the solvent was removed and solid product was collected and crystallized from ethanol.



X= OH, Cl, Br and H

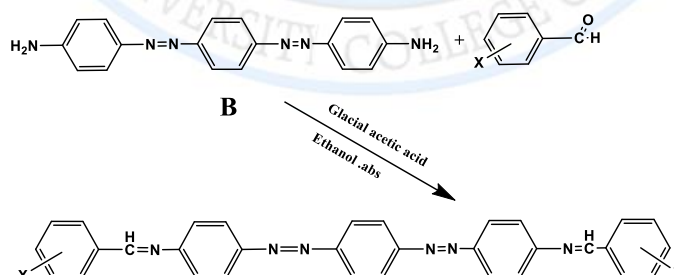
Scheme 3: the preparation (A) series compounds of Schiff base monomers

Some of the physical properties of these compounds are listed at Table (1).

Table 1: Physical properties and percentage of the monomers

					
Comp. No.	Molecular formula	X	Color	M.P(⁰ C)	Yield (%)
A ₁	C ₂₀ H ₁₅ O ₂ N ₂	Ortho -OH	Yellow	220	83.5
A ₂	C ₂₀ H ₁₅ O ₂ N ₂	Para-OH	Yellow	264	87
A ₃	C ₂₀ H ₁₅ N ₂ Cl ₂	Para-Cl	Greenish gray	212	74
A ₄	C ₂₀ H ₁₅ N ₂ Br ₂	Ortho-Br	White	150	81.7
A ₅	C ₂₀ H ₁₅ N ₂	H	White	140	79.3

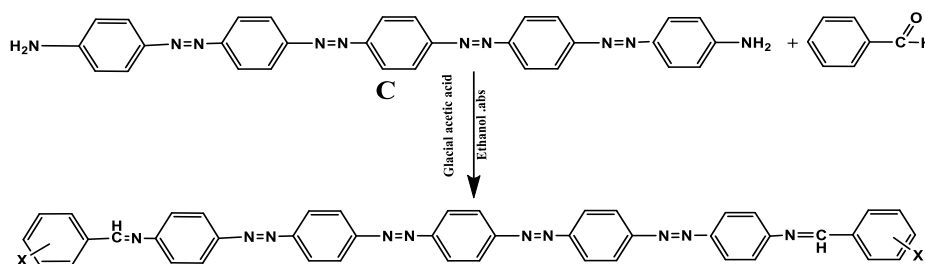
And with same method above used to Prepare Schiff base chain monomers (B₁-B₅) of (B) compound, and also to Prepare Schiff base chain monomers (C₁-C₅) of (C) compound.



X= OH, Cl, Br and H

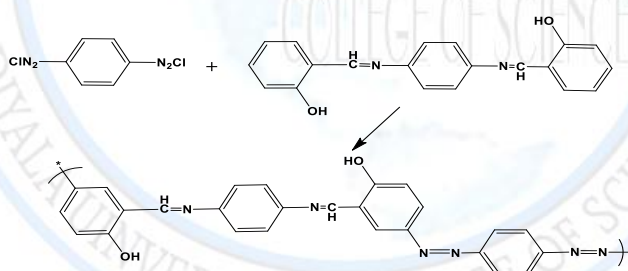
Scheme 4: the preparation (B) series compounds of Schiff base monomers

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Scheme 5: the preparation(C) series compounds of Schiff base monomers

2 ml of (Conc. HCl) with 10 ml of distilled water dissolved (0.108 g – 0.0001 mol) of (P-phenylene di amine) at (0-5⁰C), in other flask prepare solution by dissolved (0.45 g) of sodium nitrite in 5 ml of distilled water at (0-5⁰C), then added 1st to 2nd with stay temperature at (0-5⁰C) ,In 5ml of (NaOH 35%) dissolve (0.316 g – 0.0001 mol) of (A₁) at (0-5⁰C) and then added to diazonium salt with stirrer at (0-5⁰C) ,to obtained dark red precipitate.



Scheme 6: The preparation of polymers

Table 2: Instruments that used at present research

No.	symbol	Name
1-	(FTIR)	Fourier-transform infrared spectroscopy
2-	(NMR)	Nuclear Magnetic Resonance Measurement
3-	(Pom)	Hot stage polarized light microscopy.
4-	(LCR)	Electrical measurements, which include: Conductivity measurement and real dielectric.

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Results and Discussion

1. Spectroscopic Study

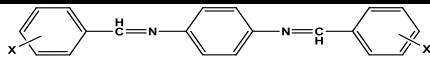
1.1: Characterize compounds (A₁-A₅) by infrared spectrum (IR)

It has been confirmed for the chemical reactions to the Schiff base by the usual way But the ratio of 1: 2 one mole of amine and two mole of aldehyde by follow-up change color as well as the study of changing physicals properties, the diagnosed of prepared compound by infrared spectroscopy (IR) as well as the Nuclear Magnetic Resonance spectrum of the proton (¹H-NMR) the studied of spectra infrared to Schiff bases prepared within the range (600-4000)cm⁻¹, two bands absorption disappearance has been observed stretchable symmetric and asymmetric of primary amine, and also the disappearance of the absorption of stretching group (C-H) aldehydes, the carbonyl group of Aldehyde disappearance and the disappearance two bands of (NH₂) of amine indication of the occurrence of reaction, it was noted the emergence of a new assertive moderate to strong intensity of azomethine group (C=N) within the range (1603-1614 cm⁻¹), and appearance the frequency of bond (C-N) in the area between (1148-1282 cm⁻¹), and also appearance bands a weak -to-moderate intensity of stretchable (C-H) aromatic within the range (3028-3057 cm⁻¹), it was frequency of stretch (C-H) aliphatic within the range (2864-2995 cm⁻¹), and the emergence of medium- intensity bands within the range (1489-1591 cm⁻¹) of stretchable band (C = C) aromatic, and band appeared at the range (683-717 cm⁻¹) to stretchable of (C-Cl), and moderate band that appeared at (654 cm⁻¹) to stretchable of (C-Br), and emergence frequency of phenolic free hydroxyl group in the prepared compounds was observed within the range (3267-3429 cm⁻¹) the form hydrogen bonding implied between the hydrogen atom belonging to the hydroxyl group and the nitrogen atom belonging to the azomethine group result to the emergence of a broad band at the lower frequencies, these bands do not show private in the ortho –hydroxy compound due to steric shape for some compound, which does not allow the formation of hydrogen bonds between the hydroxyl group and azomethine group.

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Table 3: Characteristic FTIR absorption bands of prepared monomers

Comp. No.	ν (cm) ⁻¹ , -KBr I.R 				
	C-N	C=N	C-H aromatic	C=C	other
A ₁	1192 1232	1614	3049	1576	O-H ortho 3429
A ₂	1194 1250	1612	3028	1591	O-H para 3267
A ₃	1184 1227	1610	3041	1493	C-Cl para 683-717
A ₄	1226 1261	1603	3057	1496	C-Br 654
A ₅	1184 1282	1606	3053	1572	

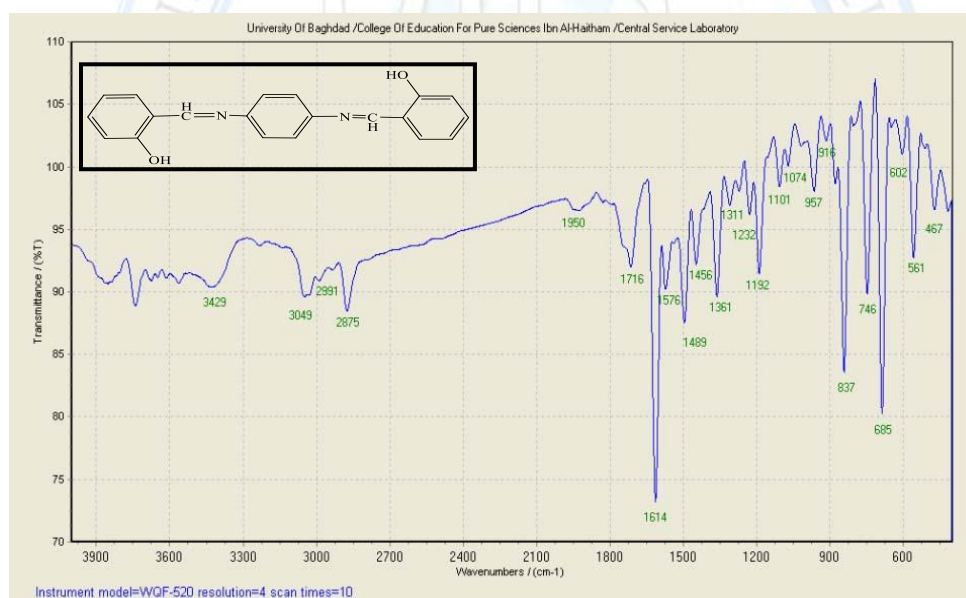


Figure 1: FTIR spectrum of (A₁) compound

1.2: ¹H-NMR spectrum of (A₅)

The ¹H-NMR (DMSO-d₆) of compound asin (figure 3) shows appeared two pair of singlet signal at (2.3)ppm attributed to solvent (DMSO-d₆) and (3.2)ppm attributed to found water with solvent ,and appeared singlet signal at (7.31)ppm that could attributed to (4) protons that type (a) of phenyl and the integration of equals (4.04) , and it observed the emergence of multi-

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signal for the result of interference between the signal at (7.43-7.53)ppm attributed to (6) protons that type (c) of phenyl, and (7.84-7.89)ppm that could be attributed to (4) protons that type of (b) of phenyl and the integration of equals (10.30) ,while the two protons of the (N=CH) group appeared at (8.63)ppm as sharp singlet signal, and the integration of equals (2)

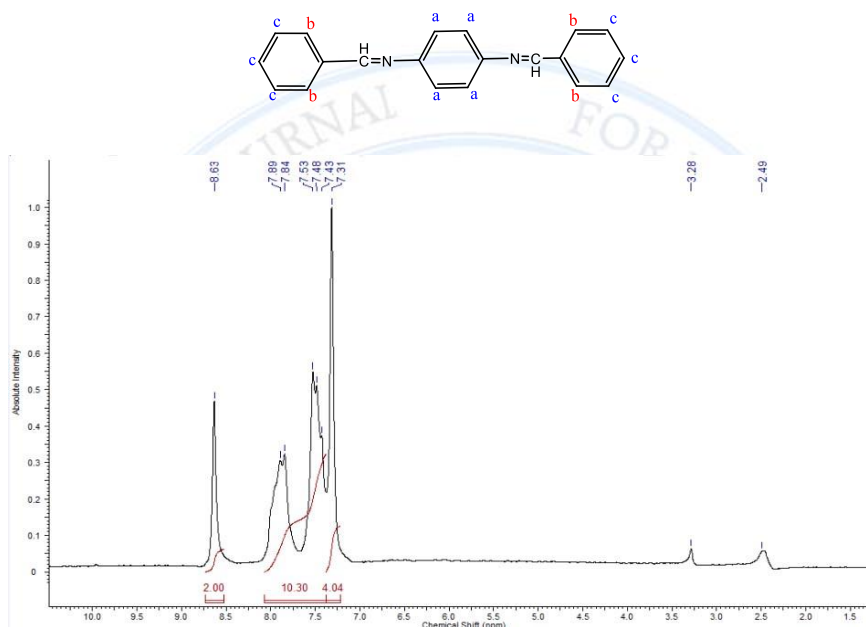
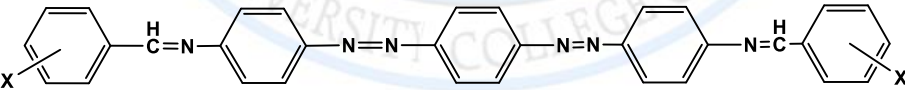


Figure 2: ^1H -NMR spectrum of (A_5) compound

Table 4: Characteristic FTIR absorption bands of prepared monomers

						
Comp No.	$\nu \text{ (cm)}^{-1}$, I.R - KBr					
	-N=N-	C-N	C=N	C-H aromatic	C=C	other
B ₁	1458	1184 1273	1653	3045	1568	O-H ortho 3392
B ₂	1504	1223 1290	1674	3060	1595	O-H para 3361
B ₃	1489	1165 1238	1697	3028	1591	C-Cl para 694
B ₄	1435 1500	1192 1261	1689	3057	1587	C-Br ortho 634.694
B ₅	1500	1165 1308	1689	3033	1591	

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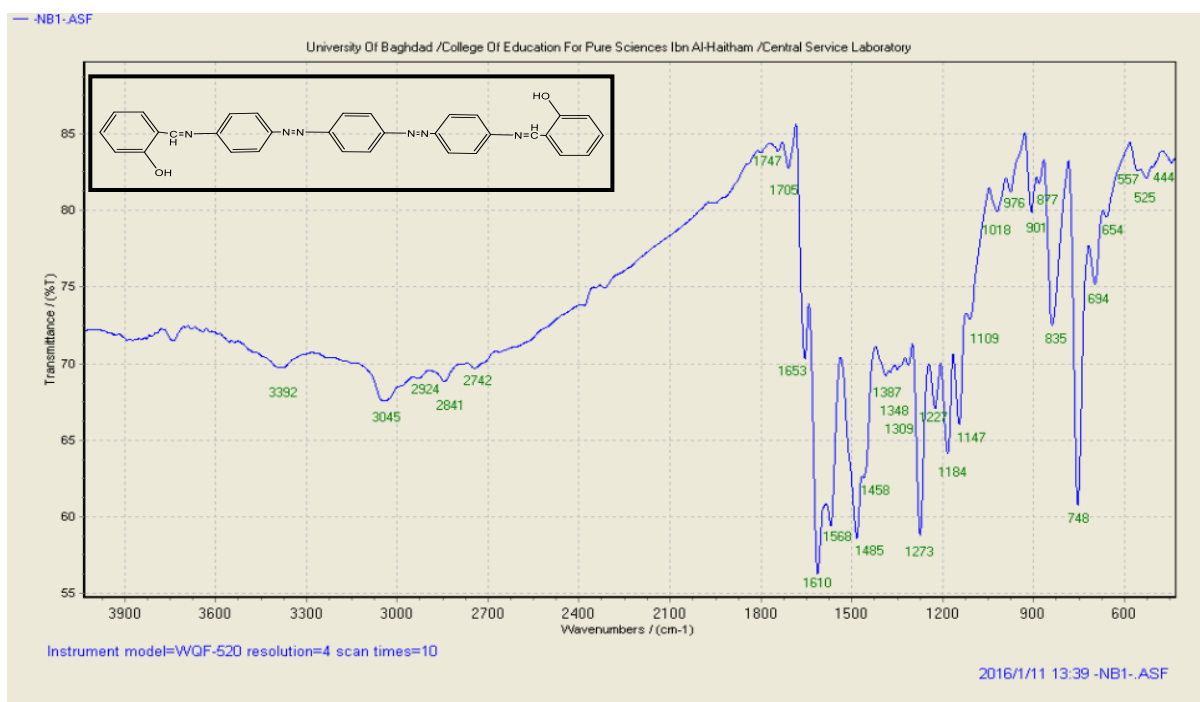
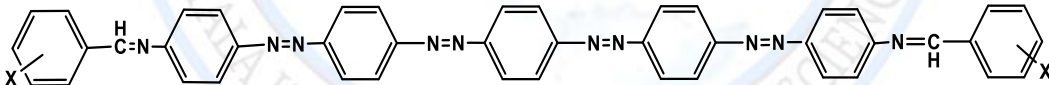


Figure 3: FTIR spectrum of (B₁) compound

Table 5: Characteristic FTIR absorption bands of prepared monomers

						
Comp. No.	ν (cm) ⁻¹ , I.R -KBr					
	-N=N-	C-N	C=N	C-H aromatic	C=C	other
C ₁	1485	1207 1277	1657	3057	1591	O-H ortho 3190
C ₂	1450	1147 1290	1682	3053	1595	O-H para 3357
C ₃	1457	1146 1287	1637	3058	1584	C-Cl para 674
C ₄	1483	1125 1223	1623	3068	1578	C-Br 688
C ₅	1493	1117 1263	1634	3074	1591	

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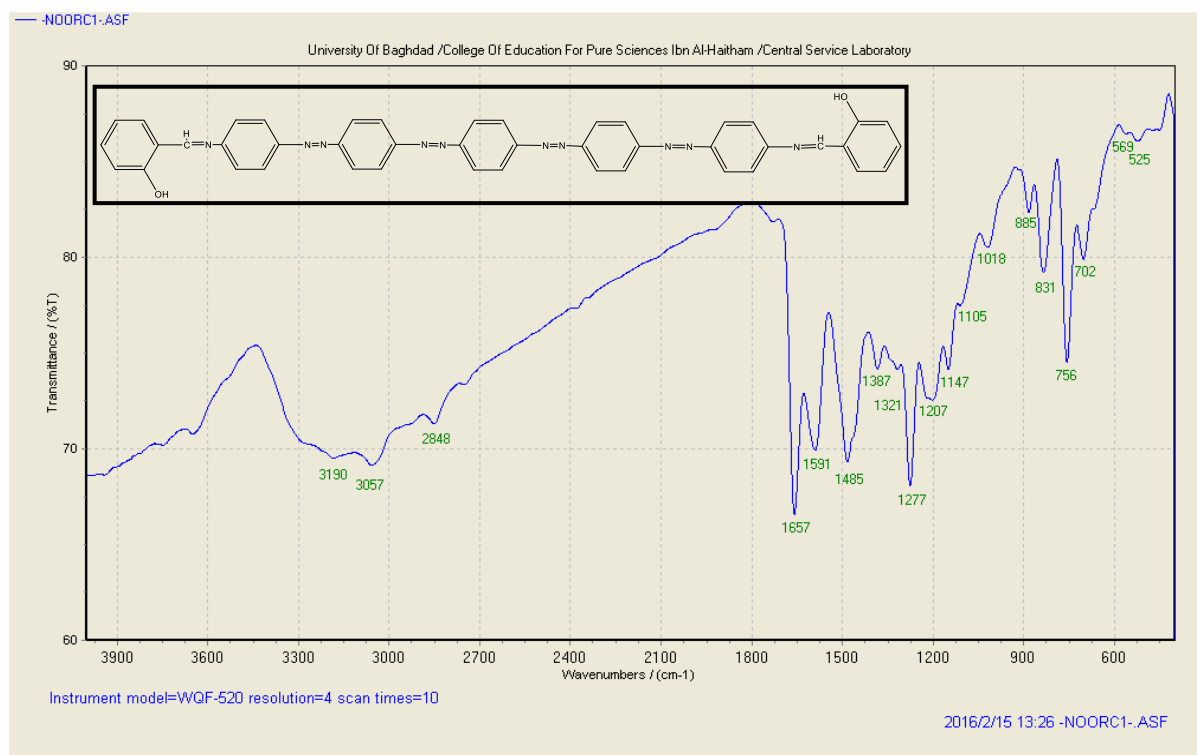


Figure 4: FTIR spectrum of (C₁) compound

Table 6: Characteristic FTIR absorption bands of prepared polymers

Comp No.	I.R -KBr , ν (cm) ⁻¹						
	-N=N-	C-N	C=N	C-H aromatic	C-H aliphatic	C=C	other
I	1494	1282	1609	3043	2995	1574	O-H ortho 4623
II	1444	1300	1616	3043	2925	1518	O-H para 3433
III	1494	1306	1638	3055	2929	1598	O-H ortho 3368
IV	1488	1276	1633	3049	2919	1601	O-H para 7433

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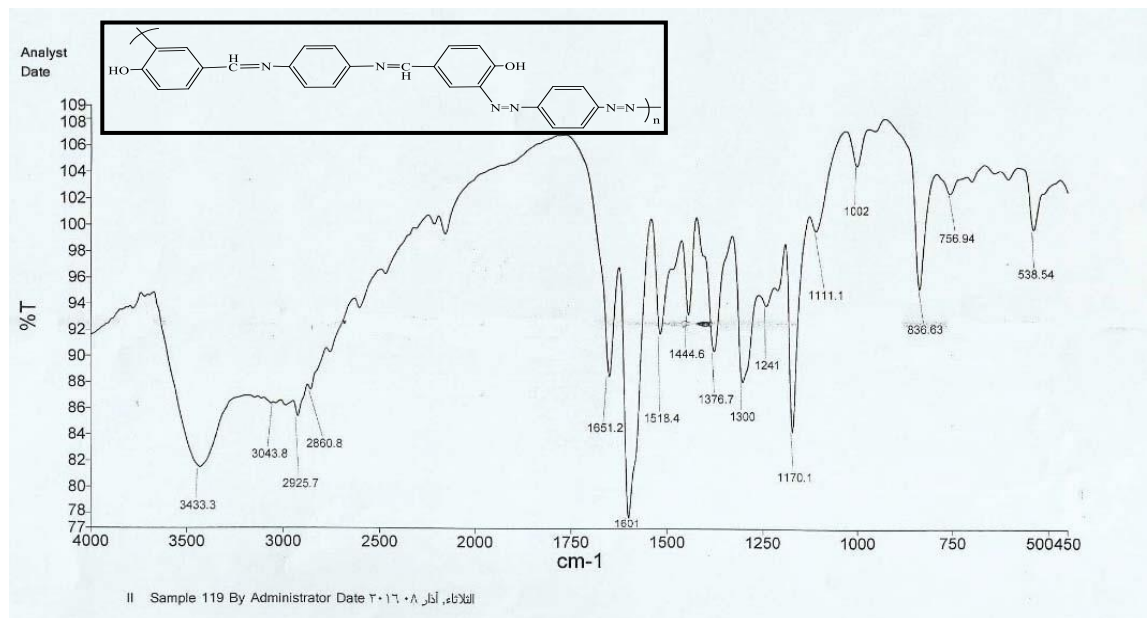


Figure 5: FTIR spectrum of (II) Polymer

2. Studying of the liquid crystalline state

Liquid crystal properties studied using hot stage polarized light microscopy, the interpret of moderation phases and the thermal stability is influenced by the nature of the community association in compounds [13] As is known, one of the most liquid crystals possess properties are linear , hardness and polarization as well as the length of the molecule to the diameter rate must be located within the range (4-6.4) Å, The reason for the interpret of the liquid crystalline phase of these prepared compounds that it possesses these characteristics as it has linear and hardness due to the presence of aggregates phenyl group which gives this character of the molecule , as well as possess polarization due to the presence of substitutes groups in the rings so some of the prepared compounds above given liquid crystalline properties , shows it possesses the ratio of the length of the average diameter within the range known As did (Teucher) and his group [14] and the two researchers (Hegee) and (Vanderveen) [15] shows Increased thermal stability because of the increasing polarization of the central molecule as a result of the electrons of double bond , Because form hydrogen bonding within the molecule,

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and does not appear liquid crystalline phase for some compounds may be due to several reasons in the forefront ratio to the average diameter of the particles prepared, it is known in the literature that the presence of hydrogen bonds interfaces cause linear crash and thus crash phase liquid crystal phase, because he raises temperature Moving as well as it encourages the emergence of non-linear structure this was confirmed in their study (Gray and Coates) [16] the interpret nematic phase of the prepared compounds (A_3 , A_5) may be interpreted on the basis that rise in temperature during the heating increases the particle's energy and thus lead to overcome the attractive forces between molecules, which allows it to arrange themselves depending on the interactions and new forces causing to be the order of molecules is parallel within the same class, between classes and thus encourage the emergence of nematic phase [17] and the following figures some histological forms of the prepared compounds at different temperatures.

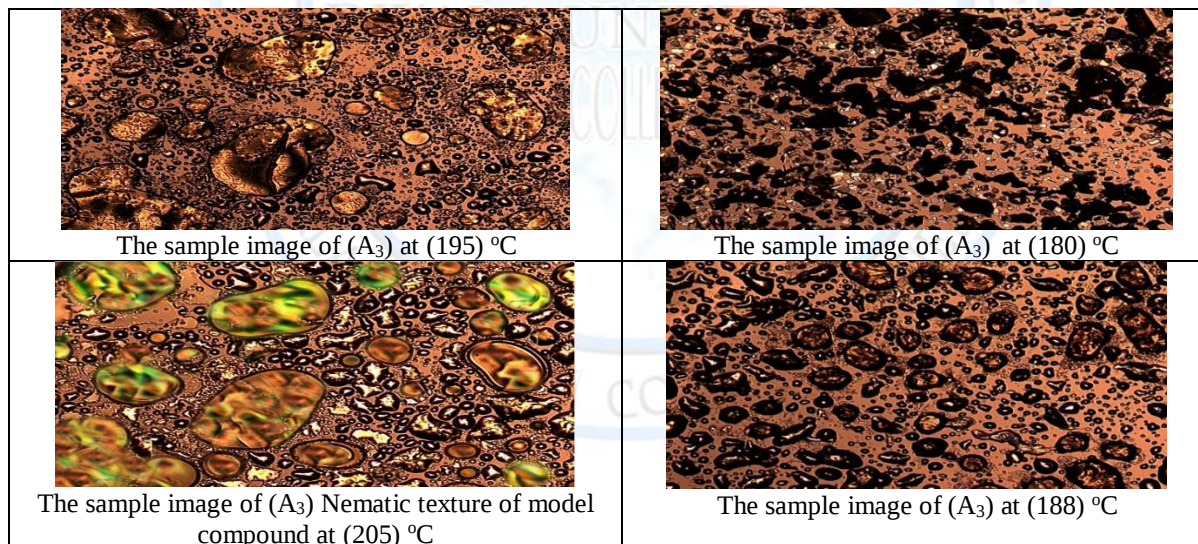


Figure 6: The sample images of (A_3) in hot stag polarized light microscopy at different temprature

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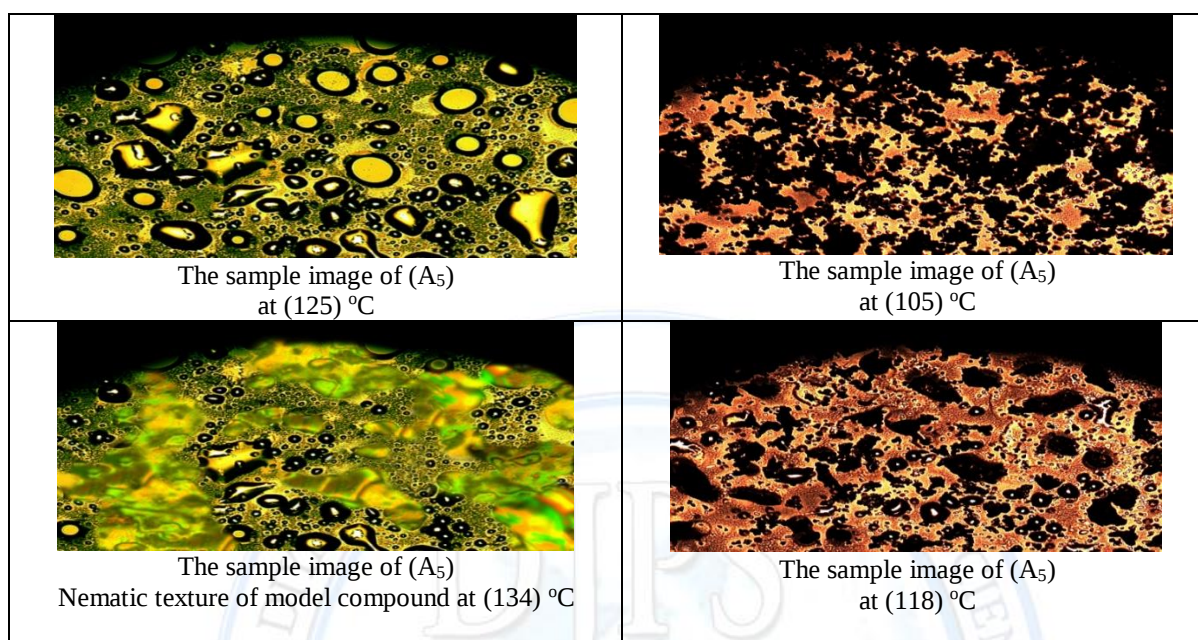


Figure 7: The sample images of (A₅) in hot stag polarized light microscopy at different temprature

3. Studying of the electrical properties

Conducted measurements of the compounds prepared using the device (LCR- 8105-G) In the laboratory service in the College of Education at the Ibn al-Haytham in that degree lab temperature by using a wide range of frequency, It was noted that changes in the conductivity values within this range(50 - 1000000 Hz),the real dielectric constant of the material measure of the ability of the material on the concentration of electrostatic field lines and also can promise (of stored electrical energy when applying the ratio of effort and longer) From the tables below were measured amplitude (F) or (faraday), the real dielectric constant can be calculated by applying the below following equation [18] , where they found the real dielectric constant values of the material prepared ,the best real dielectric constant values of the (A₁) sample are (74.2371) at (50 Hz) of frequency, while the best value of the real dielectric constant of the sample (II) is (29.21) at (5074.875) of the frequency it is the recipe for the material it possesses High real dielectric constant at this frequency, as is known in the literature , the real dielectric constant value decreases with increasing frequency, and this was identical to the results in the below tables, so it can use this material that has higher real dielectric constant as materials for

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storage energy as dilated of storage. As well as the measured conductivity of these prepared compounds by using the same frequencies, because different conjugated polymers that attended in the past 25 years with excellent electrical properties [19] , this is due to delocalization of electrons in conjugated orbitals that overlapping continuously on the backbone of the polymer chain length , Because the poly imine which is also called as the azomethine or schiff base polymers are containing double bond between carbon and nitrogen called azomethine group ($N = C$), as observed some compounds possess conductivity property and some of them were semiconducting while some of samples it found possesses insulating property, the results are described as follows in the tables below and the chart shows the conductivity relationship with frequency [20].

C: Capacity or Farad (F)

D : thickness of Disk

A : Area (Disk space)

ϵ_r : real dielectric constant

ϵ_0 : Dielectric constant imaginary $= (8.85 * 10^{-14})$

Table 7: valuable of measurement electrical of (A_1)

Unit	Hz	log Hz	$\sigma (\Omega.cm)^{-1}$	F	ϵ_r
1	50	1.69897	1.85E-06	1.25E-09	74.2371
2	5074.875	3.705425	2.43E-10	2.48E-12	0.14645
3	10099.75	4.004311	3.10E-09	2.70E-12	0.160715
4	50298.75	4.701557	3.55E-08	3.04E-12	0.181057
5	100547.5	5.002371	1.21E-08	3.77E-12	0.224418
6	201045	5.303293	7.20E-08	3.07E-12	0.182782
7	301542.5	5.479349	2.22E-07	3.13E-12	0.186113
8	402040	5.604269	1.13E-06	2.86E-12	0.170292
9	502537.5	5.701168	1.55E-07	2.82E-12	0.167793
10	603034.9	5.780342	4.47E-07	3.04E-12	0.181057
11	703532.4	5.847284	5.01E-08	3.03E-12	0.180046
12	804029.9	5.905272	4.10E-07	2.94E-12	0.174693
13	904527.4	5.956422	9.95E-08	3.00E-12	0.178202

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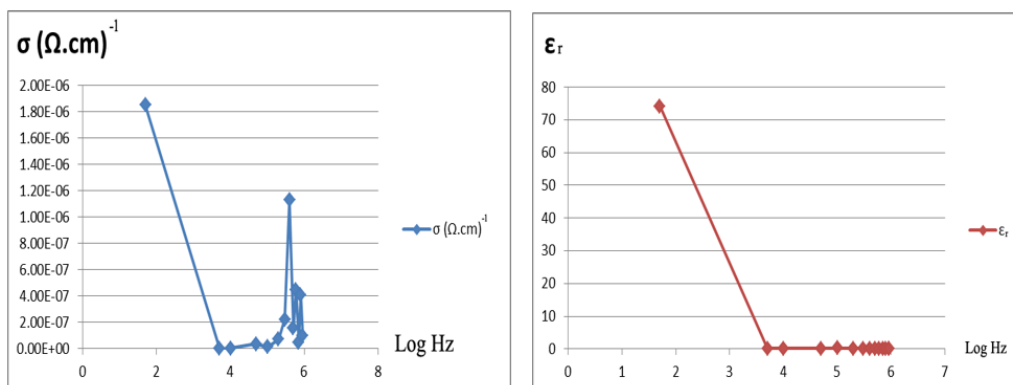


Figure 8: Relation conductivity, dielectric with frequency of (A₁) monomer

Table 8: valuable of measurement electrical of (II)

No.	Hz	log Hz	$\sigma (\Omega.cm)^{-1}$	F	ϵ_r
1	50	1.69897	2.69E-03	5.01E-10	29.809
2	5074.875	3.705425	2.58E-03	4.11E-10	24.45525
3	10099.75	4.004311	2.43E-03	2.06E-10	12.26658
4	100547.5	5.002371	2.76E-04	3.36E-11	1.999959
5	201045	5.303293	2.81E-04	2.53E-11	1.502765
6	301542.5	5.479349	2.85E-04	2.24E-11	1.334436
7	402040	5.604269	2.89E-04	2.06E-11	1.223268
8	502537.5	5.701168	2.93E-04	1.93E-11	1.14862
9	603034.9	5.780342	2.95E-04	1.82E-11	1.081824
10	703532.4	5.847284	2.97E-04	1.75E-11	1.040069
11	804029.9	5.905272	2.99E-04	1.70E-11	1.011816
12	904527.4	5.956422	3.00E-04	1.68E-11	1.001585
13	924626.4	5.965966	3.01E-04	1.68E-11	0.999563

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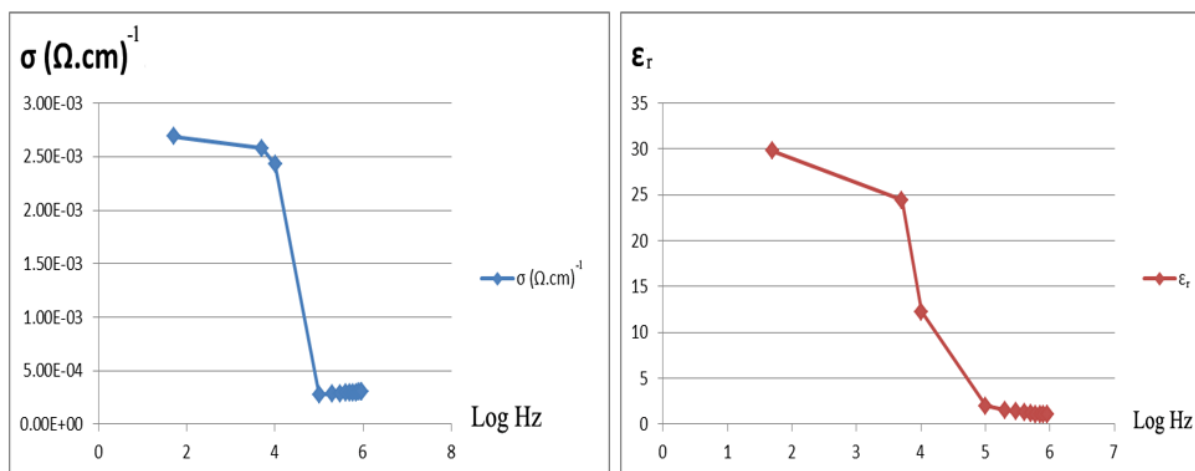


Figure 9: Relation conductivity, dielectric with frequency of (II) polymer

Conclusions

In the present work, we have successfully prepared of the schiff base compounds that has liquid crystalline properties in (Nematic phase) because they contain (H-C=N) azomethine group, which is characterized as having a liquid crystal properties, the study of the electrical properties in monomers and polymers have been prepared where the electrical conductivity at the expense of a wide range of frequency, where some of the increased conductivity values when increasing frequency while the best connectivity for some compounds at(50 Hz) ,the dielectric constant expense of monomers and polymers , it has a higher dielectric constant at (50 Hz) while decreases with increasing of frequency .

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