## Preparation of Polyacetylene Composite with some Plastic and Semiconductor for Electrical Uses.

#### Tariq Abdul-Jalil Mandeel

AL-Anbar University- College of science



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#### ABSTRACT

Three novel polyacetylenes : Polyacetylene PAs, 1-ether- benzoic acid-2- ene - propylene (A) .

Polydiacetylene (PDAs), hexa -1,6 diphthalmide - 2- ene – 4 –yne ( B ) , and Polyacetylene PAs , 1- phenyl - 3- but yne -1 -ol ( C) .

were prepared through polymerization of three Acetylinic monomers. They were prepared byby reaction of propargyl bromide, unsaturated alkyl halide RX and With, other various reagents)) Polymerization was carried out by using selective catalyst such as CuCl, PdCl2 complexed of transition metals with dry and pure oxygen . polymers were extracted , purified and identified by FT-IR, HNMR Spectroscopy and melting points . Electrical conductivity of polymers was studied after doping with iodine (I2) to increase their conductivities . Composite plastic was prepared from polymethyl metha acrylate PMMA as the matrix with Polyacetylene as the filler by cast molding the solution of plastic and polyacetylene was prepared by mixing with THF as solvent. The conductivities of the composite seem to be close to semiconductors and the results are in agreement with the literature and theoretical proposals.

#### Introduction

The conductive Polyacetylene occupied and demonstrated industrial importance .and its efficiently was proved in the industry of Solar cells, Polymers batteries and devices as parts in electronics, These polymers were prepared in different methods, including thermal polymerization, photo polymerization and radiation, (1, 2) Using catalysis of polymerization (Initiation) is similar to Zekler-Natta such as copper salts, palladium, catalyst Molybdenum, and used the Propargyl halides and their derivatives in the preparation of the types of conductive Polyacetylene as well as the preparation of Homo diacetylene & Hetro diacetylene In order to enter the terminal acetylene group easily during the reaction acetylinic monomers (3), also acetylene Polymers are used successive with manufacture of conductivity of polymers because of the Conjugation Bond type  $(\pi)$  which affected dominant factor on electrical conductivity. Aacetylinic Polymers are also used as part of the conductive such as diodes at the

\* Corresponding author at: AL-Anbar University- College of science, Iraq.

electric circuit, and chemical composition occupies of the polymers Conjugation critical uses in Applied and to its impact on Stability, and increase the thermal stability and resistance to operating conditions in electronic devices, (4, 5, 6, 7) At the present time acetylene Polymers for medical uses, such as drugs for diseases treatment that affect cancer cells is developed and used as anti- cancer besides working for the inhibition of alsaitocrom(8, 9,10) The common way to produce this type of polymers is the Hay method which uses transitional catalysis such as copperus chloride, Glaser Coupling method and Egliton method (11) use palladium, Molybdenum, and copper as complexes catalysts, of initiations polymerization, they are also used for the preparation of acetylene Copolymers, and the development of conductivity polymers with good electrical properties, though polymers have insulater characters of materials . the recent research was able to produce polyacetylene which is similar to the semiconductor or the connected metal, where conjugated polyacetylene has relatively small Energy gap (1 - 4ev). The electrons facilitates transfer to the coductivity band and gives the semiconductor its characteristics. Also they can increase conductivity of these polymers through Doping process in order to increase the oxidation state where the particles of doping are made by of increasing the movement of charge carriers which give ability of the high conductive of electricls in the Doping case (12) The common materials, used with Doping are iodine, bromine, and arsenic. are made in several methods, including electrical, chemical, complexes and the charge transfer. The double method has been used in this study, chemical doping method to increase the electrical and of the compsite conductivity properties. The advantage of conductive polyacetylene materials with high stability towards the operating conditions such temperature , humidity and environmental factors .is that it is used successfully in medicine and industry (13, 14) The present study aims preparing the conjugated polacetylene and to composited on the poly methyl metha acrylate PMMA (Plastic) for the development of the electrical conductivity properties of the insulating materials to semiconductor in order to be used in the Electrical circuits.

#### **Experimental**

#### Materials and Methodology

In this research the materials were used with of high purity, Propargyl bromide was distilled under vacuum pressure, and Cuprous chloride **CuCl** ,Palladium dichloride **PdCl**<sub>2</sub>, were used as catalysts in the polymerization processes which they were bouht (Fluka) and methyl metha Acrylate monomer (**MMA**) , Iodine **I**<sub>2</sub> (Fluka), methanol, pyridine, acetone, ether bought from (BDH) Recorded of spectra HNMR, using a BRUKER - 400.13MHZ Spectrophotometer. in CDCl<sub>3</sub>,solvent, FTIR spectra mode 18300 shimadzu spectrophotometer Japan. With (400 - 4000) cm<sup>-1</sup> and use a disc models for Potassium bromide to preparation Specimens of FTIR, these measurements were conducted in the Al-Baath University in Homs – Syria.

#### Synthesis of Monomers

#### Preparation 4 – benzoic acid propargyl ether. (a)

The reaction comprised of, **two steps first** preparation of the compound 4 - hydroxyl benzoate. of reaction **4** g of p - **hydroxyl** benzoic **acid**, 10ml of methanol was added to in flask **250** ml a circular flat bottom equipped with a condensation and separating funnel, was prepared . The mixture refluxed and stirred by a magnetic stirrer for five minutes then we add **5** ml of acetyl chloride and slowly drop wise, By continued stirring for one hour at room temperature and then stopping the reaction product was nominated and dried under vacuum pressure in the desiccator for **24** hours .

second step , dissolving 4 g of p - hydroxyl benzoat (prepared in the previous step) in 10 ml of the dimethyl sulfoxide (DMSO) with 6 g of potassium hydroxide KOH in a round flat bottom flask 250 ml, equipped with a condensation and separating funnel, for the addition. The mixture stirred by magnetic stirrer. Then was added and slowly continued was for reaction for an 1.5 hour, at room temperature then 30 ml of methanol was poured in to the reaction solution with the addition of the 4 g of sodium hydroxide (NaOH) and the product was extracted with methyl dichloride CHCl<sub>2</sub>, the product was yellowish crystals form in yield 72%, and then dried in desiccator for 20 hours, melting point was 163 C<sup>0</sup>. Monomer was characterized with spectral FT-IR.

#### Preparation N – propargyl phathalamide (b).

The monomer **b** was Prepared from reaction of Propargyl bromide with potassium phthalamide (( it was prepared by dissolving 9 g of phthalamide in 150 ml of absolute ethanol to circular flask 500 ml with flat bottom equipped with a condensationm the mixture heated in a water bath for 1 hr and then poured in to a hot solution of potassium hydroxide containing 3g\* 3ml Dist.Water,cold solution and was precipitate potassium phthalamid for treatment with acetone to removal non-reaction material, then precipitate filtrated, Colorless of potassium phthalamid)) to 5 g of potassium phthalamid dissolved in 15 ml of dimathel format( DMF) and heat the mixture to a 70  $C^0$  with stirring, then add 4 ml of Propargyl bromide drop wise then continue heating for 10 hours after it stopped the reaction and added 50 ml of distilled water after that mixture was cold recrystallize resulting in a mixture of methanol acetone and precipitate white crystals of the monomer N– propargyl phathalamide  $M.p = 151 \text{ C}^{0}$ , yield 69% . monomer characterized with spectral FT-IR.

#### Preparation 4 - phenyl - 1 - butyne - 4 - ol.(c)

2.8 ml of Propargyl bromide was dissolved in 25 ml of tetrahydrofurane (THF) and 2 gs of zinc powder was added which was also dissolved in was THF in a round bottom flask of 100 ml equipped with a condensation and separating funnel for the addition ,the mixture of Continuation with stirrers For 1.5 hr and then added 2ml of benzaldehyd was dissolved in 10 ml of THF drop wise to reaction, and after that the reaction continued for 4 hrs at temperature 30 C<sup>0</sup>, Stopped stirring and add 100 ml of a saturated solution of ammonium chloride, then we left the mixture with stirring for **0.5** hr. Filtrate the mixter to remove zinc. And then **10%** of hydrochloric acid was added then then extracted product by ether, then separated and dried organic layer with magnesium sulfate, and evaporated the solvent. The product was of yellow liquid which was characterized by FT-IR. Spectroscopy.

#### **Preparation of Polymers**

## Preparation of Polyacetylene(PAs),1-ether-benzoic acid-2-ene - propylene (A).

2.5 gs of monomer **a**, added to 5 ml DMF in Erlenmeyer flask,50 ml and 0.2 g of palladium chloride  $PdCl_2$  was dissolved in 0.5 ml of pyridine( distilled and dried over sodium metal) . The polymerization reaction was under atmosphere of nitrogen on magnatic stirrers at room temperature for 2.5 hrs. termination of the reaction by adding a few drops of methanol to precipitate the polymer .we filtrated the solution under vacuum pressure to produce white crystalline, yield 65% which is characterized with FT.IR and HNMR spectrum.

Preparation of Polydiacetylene (PDAs), hexa -1,6 diphthalmide- 2- ene – 4–yne. (B) 0.8 g of monomer b, 0.4 mg of palladium dichloride PdCl<sub>2</sub> in 7 ml of pure pyridine, and 0.1 mg of Cuprous chloride CuCl in 3 ml were prepared in erlermeyer flask of 100ml. The flask was provided with a small side tube connecting to the balloon of oxygen Store. ,the polymerization reaction was under atmospheric of oxygen ,It was then Vigorously stirrerd with high speed to ensure complete reaction . Oxygen gas had been pumped (method of Glaser ) twice in a flask reaction. The polymerization Continued for 5 hours, Few drops of methanol were added until termination of the reaction , The mixture to produce PDAs ,then recrystilization with chloroform ,white crystals of polymer at yield **76%**, which are characterized with FT.IR, HNMR.

## Preparation of Polyacetylene (PAs),- 1- phenyl - 3butyne -1 -ol,(C)

prepared Polymer C as in the same method of preparation polymer A , the yield of product 70% , and characterized by FT-IR , HNMR.

## Preparation of poly methyl metha acrylate (PMMA)

Monomer **MMA** was polymerized after washing twice with a solution of **10%** sodium hydroxide and then it was distilled under vacuum pressure ,dried over molecular sieves, to **8g** from MMA in a volumetric flask **50** mL and initiator benzoyl peroxide.**0.1%** The Polymerization was under atmosphere of nitrogen.in temperature of **70**C<sup>0</sup> for **30** minutes after that deposition of the polymer in ethanol and then dried, average molecular weight calculated (**42146**) unit by using the equation of **Mark-Hovwink** after measuring the intrinsic viscosity. <sup>(15)</sup>

#### **Doping of Acetylene Polymers**

Three polacetylenes were prepared, **A**, **B**, **C** was doping using the pure iodine within the system designed for this Purpose, which consists of two flasks connected through pipe provided for closure and opening which is linked to a pipe connected to a vacuum pump. The researched heated the iodine and allowed vapor to pass through a link of connect to a second flask containing specimens of polymer

# Preparation of composite polyacetylene (A, B, C) with poly methyl metha acrylate,

Three composites of Doping poly acetylene were prepared with poly methyl metha acrylate those; (PMMA + A), (PMMA + B), (PMMA + C)

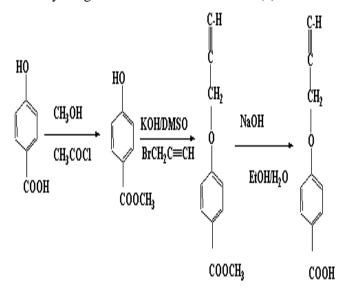
To round flask **100** ml equipped with a condensation, to 5g of PMMA was dissolved in 10 ml benzene and then added to a solution 0.2g of polyacetylene A dissolve in 10 ml benzene else, afte that heat to warm and stirring for **0.5** hr and then poured the mixture into a mold template is made of glass capacity 1 Cm x 10cm for the preparation of overlay film, and left to dry at room temperature in summer 32  $C^0$  for three hours and then cut and compression the film in a IR device specimens compressor at a pressure of 1 ton to manufacture discs 1.5 cm in diameter and thickness ranges (0.09 - 0.11) mm for the purpose of measuring Electrical conductivity. that the composites prepared in the percentages by weight 4 -100 wt% of doping polyacetylene and in the same method was Prepatedr of the other composite (**B**,**C**).

#### **Electrical measurements**

Were measured electrical conductivity of the studied composite specimens of (**PAs** + **PMMA**) using of the electrical Laboratory cell designed for this purpose scheme (8) . Was measured of volumetric connectivity to shed suitable voltage between the electrodes and then measure the current output and using the following equation:  $\sigma = IL / AV$ , where ... A = cross-sectional area cm<sup>2</sup>, V = voltage, L = length specimens in cm, I = current amps unit and electrical conductivity.

Results and discussion:

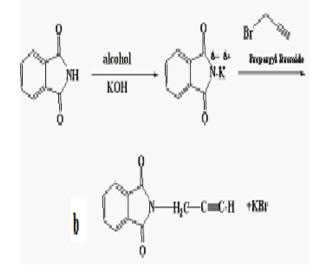
The **monomer a** prepared by the reacting of 4 hydroxy - benzoic acid, with presence of acetic chloride and methanol, the ester product in the first step of the reaction, This process included withdrawing of water molecule, then the Propargyl bromide was enters in **SN2** reaction after withdrawal of hydrogen atom in presence of strong base and **DMSO** as solvent, the ether product when the substitution reaction occurs on the oxygen atom, the ester precipitated in presence of alkaline methanol, therefore lose the acetyl group and turns back into a carboxylic again as shown in the scheme (1).



Scheme (1): the preparation of monomer (a).

Fig. (1) FT-IR spectrum of monomer show the stretching vibrations of terminal acetylene  $\equiv$  CH at 3415 cm<sup>-1</sup>, and stretching vibrations C = O at 1620 cm<sup>-1</sup> and the Bending  $\equiv$  C-H in 750 cm<sup>-1</sup> and the band is weak to C  $\equiv$  C at 2375 cm<sup>-1</sup> and bending vibrations of aromatics C = C at cm<sup>-1</sup> 1570. cm<sup>-1</sup>. fig (1).

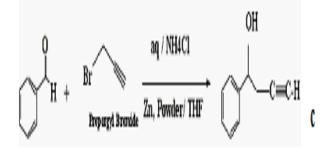
**The monomer b** prepared from the reacting of potassium phthalamide and Propargyl bromide with alcoholic potassium hydroxide there where it works to show negative charge on the phthalamide nitrogen , the nitrogen atom becomes is a strong nucleophile reagent, that can attack the carbon atom was Contains the halogen Bromide in Propargyl bromide and displacement the halogen atom to give the resulting N-Propargyl phthalamide **b**. as well as the reaction in **SN2** mechanism<sup>.(16)</sup> scheme (2) Spectra of FT-IR show stretching vibrations to  $\equiv$  CH at **3295** cm<sup>-1</sup> ,stretching vibrations to C = O, at **1720**cm<sup>-1</sup>, **1766** cm<sup>1</sup> And the bending  $\equiv$  C-H in **736** cm<sup>-1</sup> and bending vibrations to Ar -C-H at **1326** cm<sup>-1</sup> and bending vibrations of



Scheme (2): the preparation of monomer (b). C = C aromatics in cm<sup>-1</sup> 1530 cm-1 fig (2).

prepared the monomer c, scheme (3) from reaction of benzaldehyd with the Propargyl bromide in existence of zinc powder as a catalyst and solvent THF and an aqueous solution of saturated ammonium chloride, obtained the reaction with Barber mechanism to Produces a complex with zinc Propargyl to gives him the ability to push the electrons pair to attack on the electrophyl carbonyl group in the benzaldehyd, so made a new link on the carbon atom of the aldehyd group, Then Withdraw of the aromatic hydrogen from the reaction medium to and formation OH group associated with the monomer product c fig. (3) spectrum of FT - IR show peak stretch to group  $\equiv CH$ at **3295** cm<sup>-1</sup>, and the peak stretch to the group  $C \equiv C$ at 2252cm<sup>-1</sup>, peak stretch to the group OH in 3471 cm<sup>-</sup> <sup>1</sup> and stretch C = C in **1620** cm<sup>-1</sup>

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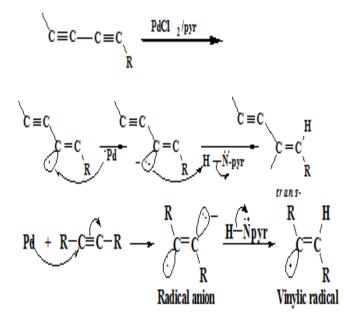


Scheme (3): the preparation of monomer c

prepared two types of polymers the first is polyacetylene PAs of the polymer (C, A) under atmosphere of pure nitrogen and palladium dichloride salts PdCl<sub>2</sub> as catalysis has disolved in DMF with presence of dry pyridine as a base. the polymerization were carried out at a temperature of **30** C<sup>0</sup> and a time did not exceed **4hrs** with strong stirring that the termenation of polymerization determined by adding few drops from methanol as precipitator. and includes of the Polymerization reaction to removal terminal hydrogen atoms from monomer by help dichlor palladium catalyst and breaking of one bonds from  $\pi$ triple bond to become hybridization than **SP**<sup>3</sup> to **SP**<sup>2</sup>. Schemes (**6**, **8**). <sup>(17)</sup>

The second type of polymer is poly daiacetylene **PDAs** of the polymer **B** was prepared by oxidative coupling method in presence of an atmosphere of dry oxygen and the reaction by transitiom element technolgy like the method of Zeklar - Natta but differ in composition and mechanism,

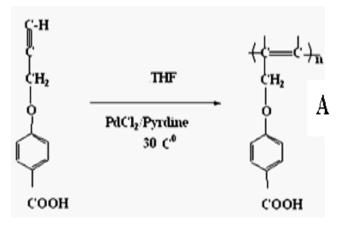
And used of palladium dichloride  $PdCl_2$ , Cuprous chloride CuCl, where hydrogen atoms are removed from the terminal acetylene by the presence of cuprous chloride as a catalyst and **THF** solvent with Glaesr Coupling method ,the second step, the palladium dichloride support the formation of polydiacetylen and the removal of the hydrogen atom from acetylene , where reduced the two triple bond to one of acetylene group to double bond as a free ion, so the role of palladium as an striped of the hydrogen atom, and gets the anti in the double bond to substitutes R group. scheme (4).



Scheme (4) Mechanism preparation of poly diacetylene

## Preparation of Polyacetylene (PAs), 1-ether-benzoic acid- 2-ene- propylene (A)

The polymer was prepared with the same method above and the polymerization time was **2.5hr** in **30**C<sup>0</sup>. after the reaction termination, the polymer was extraction from the solvent and dry under vacuum pressure for **12h**rs in the room temperature scheme (**5**). FT-IR spectra, fig. (**4**) .show a stretch vibrations C = C in **1870** cm<sup>-1</sup> of the conjugated double bonds, stretch to O- H at **3110** cm<sup>-1</sup> the carbonyl stretch C = O at the **1720** cm<sup>-1</sup>, bendenig to C = C at **1550** cm<sup>-1</sup> and the bending of Ar - H aromatic in **740** cm<sup>-1</sup> and stretch bendenig to C-H aliphatic at **16101** cm<sup>-1.</sup>

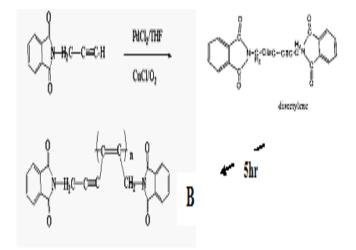


Scheme (5): the preparation of polymer A

The HNMR spectra fig (5) show that the displacement of the Ar- -CH ring in, 7.25  $\delta$ , signal – COOH at 8.26  $\delta$  signal of a group - CH2 - in 1.38  $\delta$ , signal of the two double bond to the group C = C - C = C- in 5.35  $\delta$ . and strong signal in 5.42 $\delta$  belonging to the group R - O - C-H.

## Preparation Poly-diacetylene 1,6 di phthalmide- 2 -3- dieen- 4-yne. PDAs. (B)

The Polymer **B** was Prepared by Oxidative coupling polymerization as was Previous. scheme (6) that the reaction time was **5 hrs**. and the polymer was characterized by spectrum of Infrared FT-IR. that show disappearance a peak to  $\equiv$  CH at **3295** cm<sup>-1</sup>, which represents the terminal acetylene frequencies and the appearance of successive stretch to the ethylene group (C = C) exist a weak peak **at 2167** cm<sup>-1</sup>. The vibrations of C = O at **1750** cm<sup>-1, \*</sup> and stretch to amido in **1720** cm<sup>-1</sup>, and the C  $\equiv$  C stretch with a weak peak at **2240** cm<sup>-1</sup>. Fig.(6).

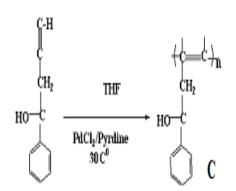


### Scheme (6) the preparation of Poly diacetylene PDAs (B).

And the HNMR spectrum fig. (7) show the following signals: the multi signals to the protons armmatic ring (7.98), (7.28)  $\delta$ , signal to group  $C = C-CH_2$  in (4.48)  $\delta$  and a signal to the conjugation of C = C - C = C - H at (5.81)  $\delta$ .

## Preparation of Polyacetylene- 1- phenyl - 3- butyne -1 -ol, ( C)

Prepared polymer C in the same way as the preparation of polymer A in scheme (7). Time of the reaction was 2hrs. Polymer extracted and dried as in the Previous routes of polymerization. and Characterized by FT-IR spectroscopy the fig.(8) show the following peak, disappearance of peak  $\equiv$  C-H at frequency in **3210** cm<sup>-1</sup>, stretch to a C- C = C- C = C in **1900** cm<sup>-1</sup>, peak in the **3180** cm<sup>-1</sup> to OH and peak = C - H in **3020** cm<sup>-1</sup>, stretch of bending to group for a C-C in 1250  $\text{cm}^{-1}$ , the HNMR spectrum fig. (9) show signal to ethylene C = C - C = C-H at 5.2  $\delta$ , signal at 4.3  $\delta$  belonging to the OH, multiple signal to aromatic hydrogen atoms of Ar – H ring in 7.43  $\delta$ and the signal at 2.51  $\delta$  to methyl group - CH<sub>2</sub> - .



Scheme (7) the preparation of polymer (C)

#### **Electrical Conductivity Measurement of Polymers**

Electrical conductivity of composite polymes (MC, MB, MA) was calculated. It was prepared from conjugated PAs with PMMA in the percentage weight (4 - 100) Wt%, see table (1). The calculated values of electrical conductivity of PMMA 1.  $2 \times 10$ -17<sup>-1</sup> scm<sup>-1</sup> in the lowest values were given in the laboratory circuit (8). This means the activation energy is high to allow electron transference between parity band and conductivity band due to the large value to energy gap. The energy required for electronic transition in plastic (insulators) is much high. and the high values in the electrical conductivity of the composites are MB compared with the composite MA and MC because the conjugation between the triple bond and the double bond in polydiacetylen (MB) increases in connectivity and it is affected on accelerating the movement of electrons through  $\pi$ bonds, The electrical conductivity increases when the percentage values increase in the polyacetylene as in a valuable 80% in composite MB.

#### **Conclusions:**

- Three terminal acetylene monomers were prepared by organic reactions from kind SN2 .
- 2 New polyacetylene PAs and polydiacetylene PDAs were prepared that have possible ability of

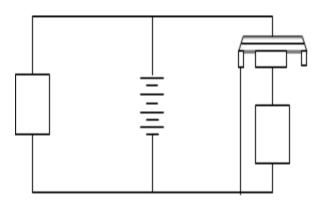
semiconductor through electronic conjucation to give the highest values of electrical conductivity.

- 3 The present study was able to develop the electrical properties from insulators to semiconductors.
- 4 The methods of oxidative coupling succeeded in preparing polyacetylene in a short and cheap method.

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  Plymer chemistry vol, 45 999 100 (2007)



Scheme (8) circuit of used to measure the conductivity

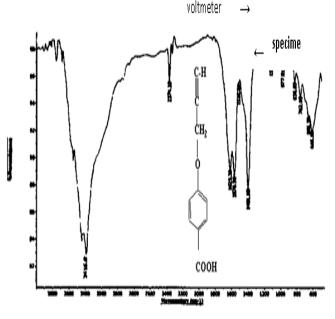


Fig. (1) FT – IR Spectrum of monomer (a)

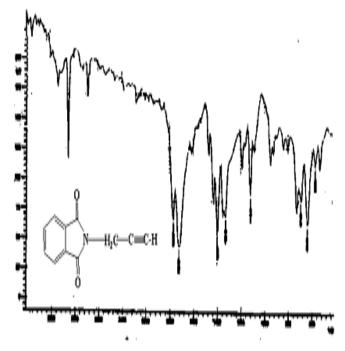


Fig. (2) FT – IR Spectrum of monomer (b)

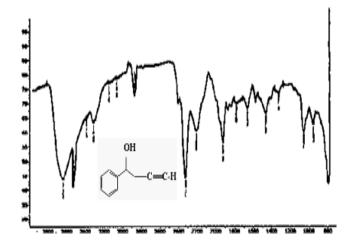


Fig. (3) FT – IR Spectrum of monomer (c)

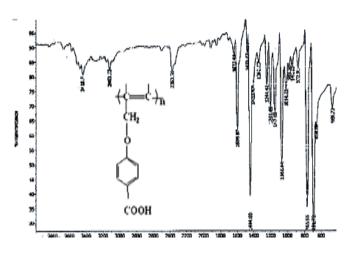


Fig. (4) FT – IR Spectrum of Polymer (A)

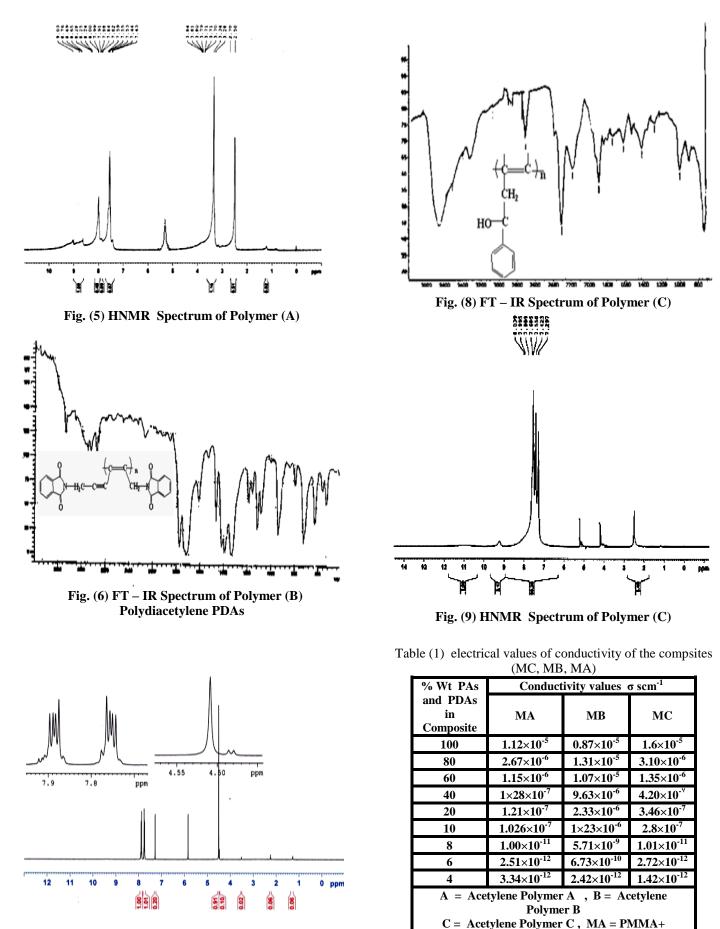


Fig. (7) HNMR Spectrum of Polymer (B) Polydiacetylene PDAs

Composite polyacetylen

## تحضير بوليمرات أستلينية متراكبة مع بعض أنواع البلأستك وشبه موصلة للأستخدامات الكهربائية

#### طارق عبدالجليل منديل

الخلاصة:

تم في هذا البحث تحضير ثلاث بوليمرات أستلينية جديدة هي :-

Polyacetylene PAs, 1-ether- benzoic acid-2- ene - propylene (A) . Polydiacetylene (PDAs), hexa -1,6 diphthalmide- 2- ene – 4 –yne. (B). Polyacetylene PAs, 1- phenyl - 3- butyne -1 -ol, (C). من بلمرة ثلاث مونومرات أستلينية تم تحضيرها من تفاعل بروميد البروبارجيل مع جزيئات مختلفة. أجريت عمليات البلمرة بأستخدام عوامل مساعدة انتقائية هي أملاح العناصر الأنتقالية مثل CuCl، PdCl2. وHNMR وFT-IR و

دراسة التوصيل الكهربائي للبوليمرات بعد تشويبها باليود (I2) لزيادة قابلية التوصيل الكهربائي. وحضرت متراكبات من البوليمرات الأستلينية المحملة على البولى ميثايل ميثا أكريليت PMMA. وكانت قيم التوصيلية المحسوبة مقاربة لأشباه الموصلات. والنتائج متفقة مع الأدبيات والأفتراظات النظرية.