A Study of Synthesis and Characterization of New Aromatic Poly(amide-imide)s

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Abstract

New thermally stable aromatic poly(amide-imide)s (PAI^1- PAI^4) were synthesized from direct polycondensation reaction of Terephthalic acid and Phthalic acid with two new different diamine monomers derivatives of 1,2,4,5-tetracarboxilic benzene dianhydride as a second diacides in a medium consisting of triphenyl phosphite (TPP) in N-methyl-2-pyrrolidone (NMP) / pyridine solution containing dissolved calcium chloride $CaCl_2$. The polymerization reaction produced a series of novel poly(amide-imide) in high yield. The new monomers were characterized by FTIR, ^{1}H-NMR spectroscopy. The resulting polymers were typically characterized by means of FT-IR, ^{1}H-NMR spectroscopy, and solubility tests. Thermal properties of the poly(amide-imide)s were also investigated using differential scanning calorimetry (DSC). Data obtained by thermal analysis revealed that these polymers showed very good thermal stability. These polymers were readily soluble in various organic solvents**.**

Key Words :(Aromatic Poly amides , Aromatic Poly imides 1,2,4,5-benzene tetracarboxylic dianhydride).

1. Introduction

Polymers play an important role in the development of materials for industrial applications such as biochemical science and in the pharmaceutical industry due to their exceptional properties[1]. They are relatively inexpensive, can be functionalized to achieve required optical, electronic, or mechanical properties, and have demonstrated compatibility with various patterning methods[2].

Aromatic polyimides have high thermal stability, good mechanical properties, and good chemical resistance, but because they soften or melt at high temperatures and have poor solubility in common organic solvents, they have low processability and limited use[3,4]. To overcome these drawbacks, many investigations have focused on modification of their chemical structure; for example, through the introduction of flexible linkages, asymmetric units, or bulky substituents into their polymer backbones[5-10]. In addition, various copolyimides, such as poly(amide-imide)s, have been proposed by many researchers[11-14]. There is a growing interest in poly(amide-imide)s (PAIs) for a variety of applications, as they retain good mechanical properties at high temperatures and are more processable than other aromatic thermo stable polymers, such as polyamides and polyimides[15].

Poly(amide-imide)s are useful high-performance materials that exhibit better processing characteristic with polyimide of analogous structures[16,17]. They find numerous applications in adhesives, electronic wire enamel, injection molding, extrusion products and membranes[18-21]. Poly(amide-imide)s thermoplastics have been the material of choice for the fabrication of large load bearing composites due to their high strength, superior stiffness, excellent thermal resistance, and high performance. Poly(amide-imide) has been extensively used in filtration media, wire enamels and for reinforcement by virtue of its superior chemical resistance and physical properties at higher temperature[22].

In our previous reports, we described the synthesis and characterization of new different poly(amide-imide)s that contain phenylene rings and flexible methylene units in the main chain, with improved solubility and thermal properties[23-25].

2. Experimental

2.1 Instruments

Fourier transform infrared (FTIR) spectra were recorded on a SHIMADZU-FTIR-8400S spectrometer (Japan) with KBr pellets in the optical range of 400–4000cm^{-1}. ^{1}H NMR spectra were registered using a Bruker, 250Mhz, spectrometer, at polymer laboratories Co Iran using DMSO as a solvent. Differential scanning calorimetry (DSC) was measured by DSC 131 Evo, SETARAM,(France) using a heating rate of 10°C/min in N_2 atmosphere within the temperature range of 30–600°C. The sample weight used approximately (10mg) . The peaks are used to determine the thermal properties of the samples .The solubility of the polymers was determined with (0.01*gm*) of poly(amide-imide) in (2*ml*) of a solvent.

2.2 Materials

N-methyl-2-Pyrrolydinone (NMP) from (ALFA-PRODUCTS); Absolute Methanol, Lithium Chloride, all from (BDH/England); Dichloromethane from (BIOSOLVE); 1,4-phenylene diamine, 4,4-diamino diphenyl methane all from (CHEM-SUPPLY); Diethyl ether from (IGCC /England); Dimethyl Sulphoxide (DMSO), m-Cresol, N,N-Dimethylacetamide, Phthalic acid, Terephthalic acid, Triphenyl phosphite (TPP), Tetrahydro- furane (THF) , all from (MERCK); Absolute Ethanol from (Scharlab S.L); 1,2,4,5-benzene tetracarboxylic dianhydride (BTDA), Isoquinoline, Toluene all from (ALDRICH); N,N-Dimethylformamide (DMF) from (Sinopharm Chemical Reagent).

2.3 Synthesis of Monomers

2.3.1Synthesis of 2,6-bis(4-(4-aminobenzyl)phenyl)pyrrolo[3,4-*f*]isonidol-1,3,5,7(2H,6H)-tetraone [M1].

1,2,4,5-benzene tetracarboxylic acid dianhydride (0.88gm / 4mmol) was dissolved in 10 ml of m-Cresol in a 150ml flask, equipped with N_2 inlet and (1.6gm / 8mmol) of 4,4-diaminodiphenylmethane was added to it in one portion. To this mixture 1ml of Isoquinoline and 45ml of Toluene were then added, and refluxed for about (5 hrs) . The water thus formed was distilled off azeotropically in a dean stark trap. Heating was continued to distill off the residual toluene. After cooling, the solution was poured into excess methanol with vigorous stirring and the precipitated product was isolated by filtration and dried in vacuum at 90°C for (24 hrs) to give 1.85gm (80%*wt*) of brown crystals. m.p = 300°C/decomposition**.**

2.3.1 Synthesis of 2,6-bis (4-aminophenyl)pyrrolo[3,4-*f*]isonidol-1,3,5,7(2H,6H)-tetraone [M2].

This monomer was synthesize by above procedure using 1,2,4,5-benzene tetracarboxylic acid dianhydride (0.88gm / 4mmol) with (0.88gm / 8mmol) of 1,4-phenylene diamine to give 1.53gm (90%*wt*) of deep brown crystals. m.p = 275°C/decomposition.

2.4 Synthesis of poly(amide-imide) (PAI^1- PAI^4):

A 500ml four neck round flask equipped with a nitrogen inlet, a thermometer, and a mechanical stirrer was charged with (0.083gm / 0.5mmol) of diacid, (0.5mmol) of diamine monomer, that shows in (Table 1), (3gm) of LiCl, (10ml) of Pyridine, (1ml) of TPP, and (5ml) of NMP. The mixture was heated under nitrogen to 110°C and stirred for 3h. The viscous solution was then cooled down and precipitated into (50ml) of methanol. The stringy polymer was washed thoroughly with hot water and methanol, collected by filtration, and dried at 100°C under vacuum.

3. Results and Discussion

3.1 Synthesis of Monomers

3.1.1 Synthesis of [M1].

M1is prepared by the condensation of one molecule of 1,2,4,5-benzene tetracarboxylic acid dianhydride with two molecules of 4,4-diaminodiphenylmethane in the presence of Isoquinoline in Toluene as catalyst in m-Cresol as solvent at 120°C for 5hs. This monomer was characterized by FTIR

Characterization of [M1] :- FTIR Spectrum

(Figure 1) which indicated absorption bands at ($3479cm^{-1}$) to (–NH_2 group), ($3217cm^{-1}$) to (aromatic –CH stretching), ($2908cm^{-1}$) to (aliphatic –CH stretching), absorption bands around ($1620–1512cm^{-1}$) show the presence of the aromatic ring, and the sharp band at ($1620cm^{-1}$) to (C=O amide). Characteristic absorption of (C=0 anhydride) disappeared.

^{1}H-NMR spectrum of (M1), is shown in Figure 2 assigns the following chemical shifts; δ (2.5) ppm for DMSO ,δ (3.7- 4.3) ppm (s,2H) for CH_2, δ (5.5) (s ,2H) for NH_2 group , δ (6 - 8.9) ppm (s, 18H) for Ar-H group .

3.1.2 Synthesis of [M2].

M2 is prepared by the condensation of one molecule of 1,2,4,5-benzene tetracarboxylic acid dianhydride with two molecules of 1,4-phenylene diamine in the presence of Isoquinoline in Toluene as catalyst in m-Cresol as solvent at 120°C for 5hs. This monomer was characterized by FTIR.

Characterization of [M2] :- FTIR Spectrum :

(Figure 2) which indicated absorption bands at (3463cm^{-1}) to (–NH_2 group), (3232cm^{-1}) to (aromatic –CH stretching), absorption bands around (1620–1512cm^{-1}) show the presence of the aromatic ring, and the sharp band at (1620cm^{-1}) to (C=O amide). Characteristic absorption of (C=0 anhydride) disappeared.

^{1}H-NMR spectrum of (M2), is shown in Figure 3 assigns the following chemical shifts; δ (2.5-3.3) ppm for DMSO , δ (6.3) (s ,2H) for NH_2 group , δ (7.6-8.5) ppm (s, 12H) for Ar-H group

3.2 Synthesis of poly(amide-imide) (PAI1- PAI4)

In this section, we used polycondensation method for synthesis of a series of four new poly(amide-imide)s from the direct polycondensation reaction of Terephthalic acid and Phthalic acid with two new different aromatic diamines [M1, M2], respectively, by using Lithium Chloride (LiCl); TPP and pyridine as condensing agents in NMP as solvent. These poly(amide-imide)s have a soft segment such as methylene units in main chain for improving solubility in organic solvents in compared to aromatic poly(amide-imide)s.
These poly(amide-imide)s were characterized by FTIR and ^{1}H-NMR spectra.

Characterization of poly(amide-imide)s (PAI1- PAI4)

FTIR Spectrum of PAI1:-

(Figure 4) showed FTIR spectrum which indicated absorption bands at (3201cm^{-1}) to (aromatic –CH stretching), (2923cm^{-1}) to (aliphatic–CH stretching), absorption bands **around** (1612–1512cm^{-1}) show the presence of the aromatic ring, the sharp band at (1612cm^{-1}) to (C=O amide).

FTIR Spectrum of PAI2

(Figure 5) showed FTIR spectrum which indicated absorption bands at (3111cm^{-1}) to (aromatic –CH stretching), (2916cm^{-1}) to (aliphatic–CH stretching), absorption bands around (1612–1512cm^{-1}) show the presence of the aromatic ring, the sharp band at (1612cm^{-1}) to (C=O amide).

FTIR Spectrum of PAI3

(Figure 6) showed FTIR spectrum which indicated absorption bands at (3224cm^{-1}) to (aromatic –CH stretching), absorption bands around (1612–1519cm^{-1}) show the presence of the aromatic ring, the sharp band at (1612cm^{-1}) to (C=O amide).

FTIR Spectrum of PAI4

(Figure 7) showed FTIR spectrum which indicated absorption bands at (3232cm^{-1}) to (aromatic –CH stretching), absorption bands around (1612–1519cm^{-1}) show the presence of the aromatic ring, the sharp band at (1612cm^{-1}) to (C=O amide).

3.3 Differential Scanning Calorimetry Study

The glass transition temperature (Tg) determined in the second heating runs of DSC measurements of the investigated poly(amide-imide)s is in range (220-242 ^{0}C). Presence of more than one endotherms in the thermograms revealed that the polymers undergo more than one phase transition processes when subjected to thermal treatment[26]. the DSC curves are shown in figures (8-11).The Tg values of Pa-i1 and Pa-i2 exhibit a linear decrease with the increase in the length of the long flexible chains, which increases the flexibility of the polymer backbone and thus the chain mobility. The polymers Pa-i1 and Pa-i2 have the lowest thermal stability than the other polymers containing phenylene moieties. This behavior can be explained by the presence of methylene units which are more vulnerable to thermo-oxidative processes [27-29]

The melting temperature (Tm) is higher fore Pa-i3 and Pa-i4 as the polymer chain more rigid parts and / or a polymer contains less free volume . Samples usually showed multiple endotherms which are explained as due to the fusion of different population of crystallites with different sizes . As expected , Tm values are lower for Pa-i1 and Pa-i2 due to the higher chain flexibility of the formers afforded with the increase the amount of methylene of polymeric back bone can influence Tm of resultant polymers. The decrease in Tm for polymer Pa-i2 than the other polymers can be explained by the presence of methylene units , in addition to the non linear phthalic acid moieties incorporated into the polymer back bone during synthesis of poly(amide-imide)s. The Tg, Tm and Tc obtained from DSC are reported in Table (2). .

3.4 Solubility of Poly(amide-imide)s (PAI1- PAI4)

Solubility of poly(amide-imide)s (PAI1- PAI4) was qualitatively tested in organic solvents and the results are summarized in (Table 3). The method that attempt to enhance their processabilities and solubilities was either by introducing bulky groups, flexible linkages, or molecular asymmetry into the polymer backbones. In this work, the attachment of bulky pendant groups in polymer backbone not only could provide an enhanced solubility because of decreased packing density and crystallinity, but also could impart an increase in Tg by restricting the segmental mobility[30].

One of the major objectives of this work was producing poly(amide-imide)s with improved solubility. The Solubility was investigated as (0.02gm) of polymeric sample in (5ml) of a solvent. All of the newly synthesized poly(amide-imide)s have good soluble in common polar and dipolar aprotic solvents without need for heating.

Conclusions

A series of aromatic poly(amide-imide)s including Pa- i1 , Pa-i2 ,Pa-i3 and Pa-i4 were synthesized by using Terephthalic acid and Phthalic acid with two new different diamine monomers derivatives of 1,2,4,5-tetracarboxilic benzene dianhydride as a second diacides respectively. FTIR and proton nuclear magnetic resonance spectra confirmed the chemical structure of the prepared poly(amide-imide)s. These poly (amide-imide)s exhibited excellent solubility in various organic solvents. The prepared polymers possess good thermal stability, and light color. Thus, these poly (amide-imide)s could be considered as new processable high-performance polymeric materials.

The Tg values of the poly (amide-imides) exhibit a linear decrease with the increase in the length of the long flexible chains, which increases the flexibility of the polymer backbone and thus the chain mobility. The polymers Pa-i1 and Pa-i2 have the lowest thermal stability than the other polymers containing phenylene moieties. The higher in Tg of (Pa-i3 and Pa-i4) compared to that analogous poly(amide-imide)s might be endorsed to the existence of phenylene moieties, in addition to the linear terephthalic acid moieties.

The melting temperature (Tm) is higher for Pa-i3 and Pa-i4 as the polymer chain more rigid parts and / or a polymer contains less free volume. Tm values are lower for Pa-i1 and Pa-i2 due to the higher chain flexibility of the formers afforded with the increase the amount of methylene of polymeric back bone can influence Tm of resultant polymers.

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Table No. (1): Synthesis of poly(amide-imide)s (PAI1- PAI4)

poly(amide-imide)	**Monomers**		**Diamine Gm**	**Yield (%wt)**	**Color**
	diacid	**diamine**			
PAI1	Terephthalic acid	M1	0.289	81	light red
PAI2	Phthalic acid	M1	0.289	83	light yellow
PAI3	Terephthalic acid	M2	0.200	83	deep brown
PAI4	Phthalic acid	M2	0.200	82	brown

Table No. (2): The glass transition, melting temperature and crystallization temperature obtained from DSC.

Sample	Tm (^{0}c)	Tg (^{0}c)	Tc(^{0}c)
Pa-i_1	368	238	250
Pa-i_2	362	220	238
Pa-i_3	502	242	266
Pa-i_4	490	240	260

Table No. (3) : Solubility of poly(amide-imide)s (PAI1- PAI4)

Solvent	Poly(amide-imide)s			
	PAI1	PAI2	PAI3	PAI4
DMAc	+ +	+ +	+ +	+ +
DMF	+ +	+ -	+ +	+ -
NMP	+ -	+ -	+ -	+ +
Pyridine	+ -	+ +	+ +	+ +
m-Cresol	+ + +	+ + +	+ +	+ +
THF	+ +	+ +	+ -	+ -
$CHCl_3$	+ +	+ -	+ +	+ -
CH_2Cl_2	+ +	+ + +	+ +	+ -
DMSO	+++	+ + +	+ +	+ +
Conc. H_2SO_4	+ +	+ -	+ +	+ +

Full Soluble. +++   Soluble at room.T. ++   Partially Soluble. + -

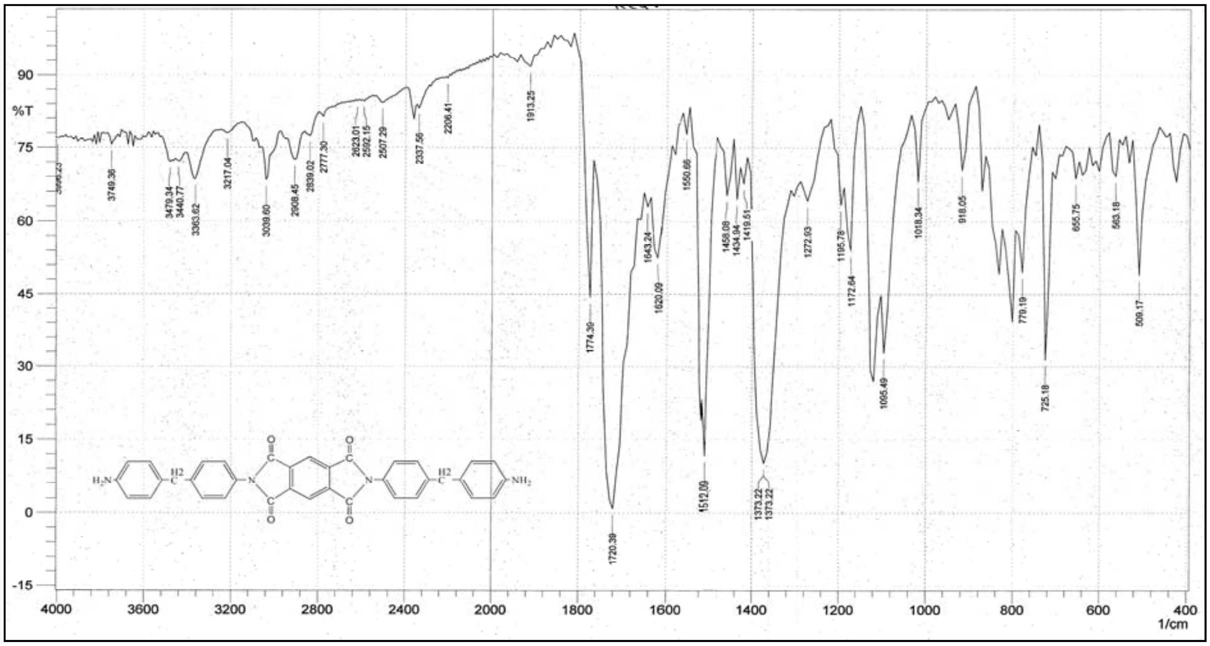


Figure No. (1) : FTIR spectrum of (M1).

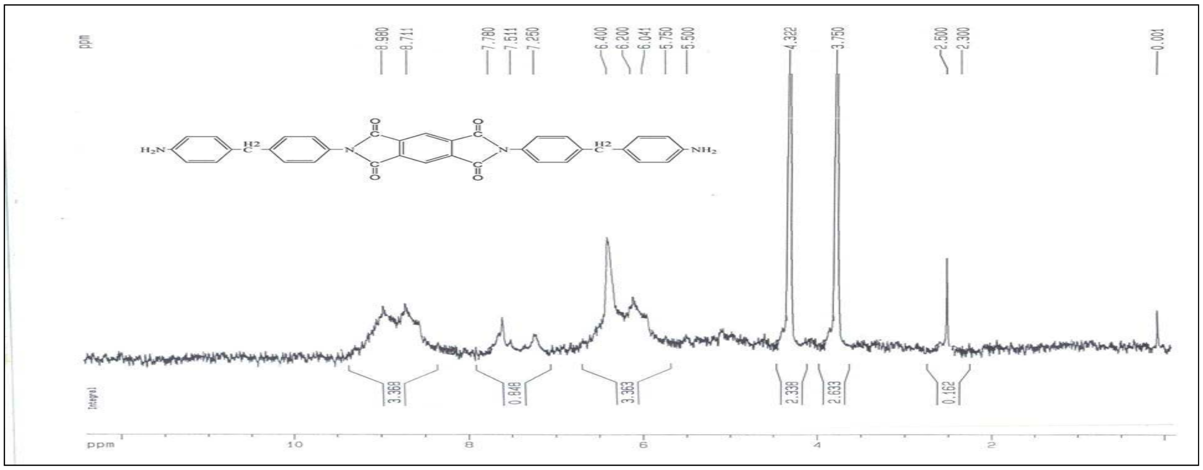


Figure No. (2):^{1}H-NMR spectrum of (M1)

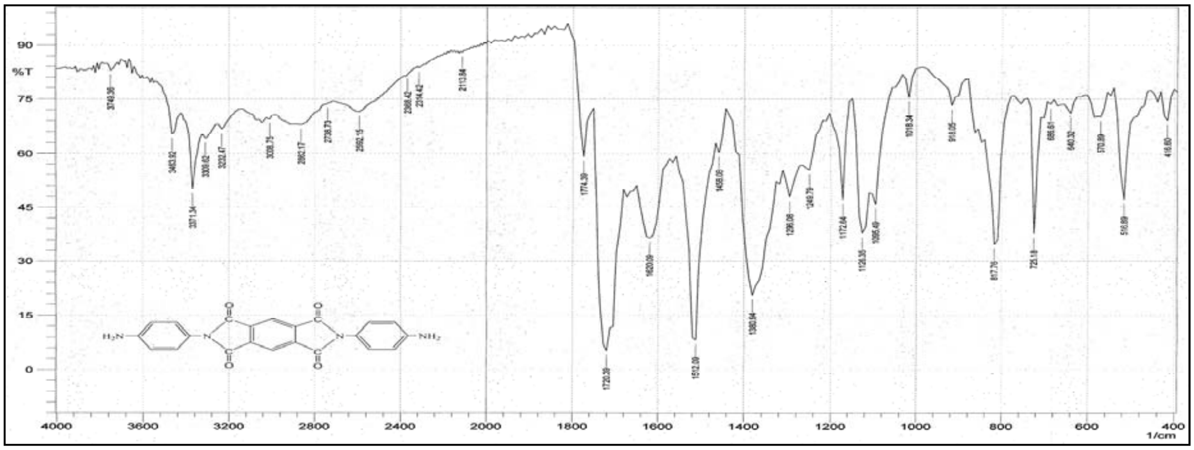


Figure No.(2): FTIR spectrum of (M2).

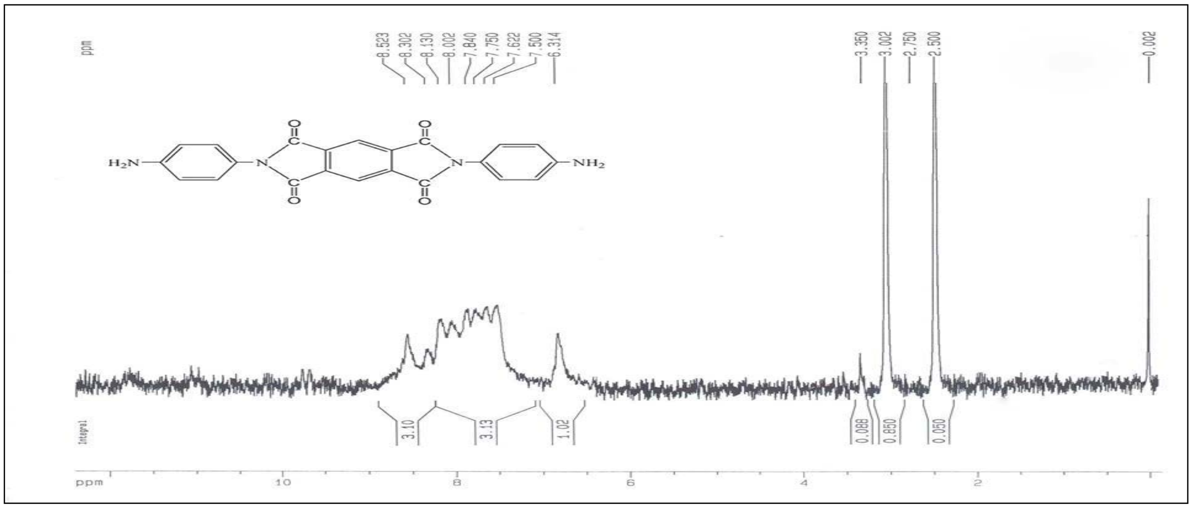


Figure No. (3): ^{1}H-NMR spectrum of (M2)

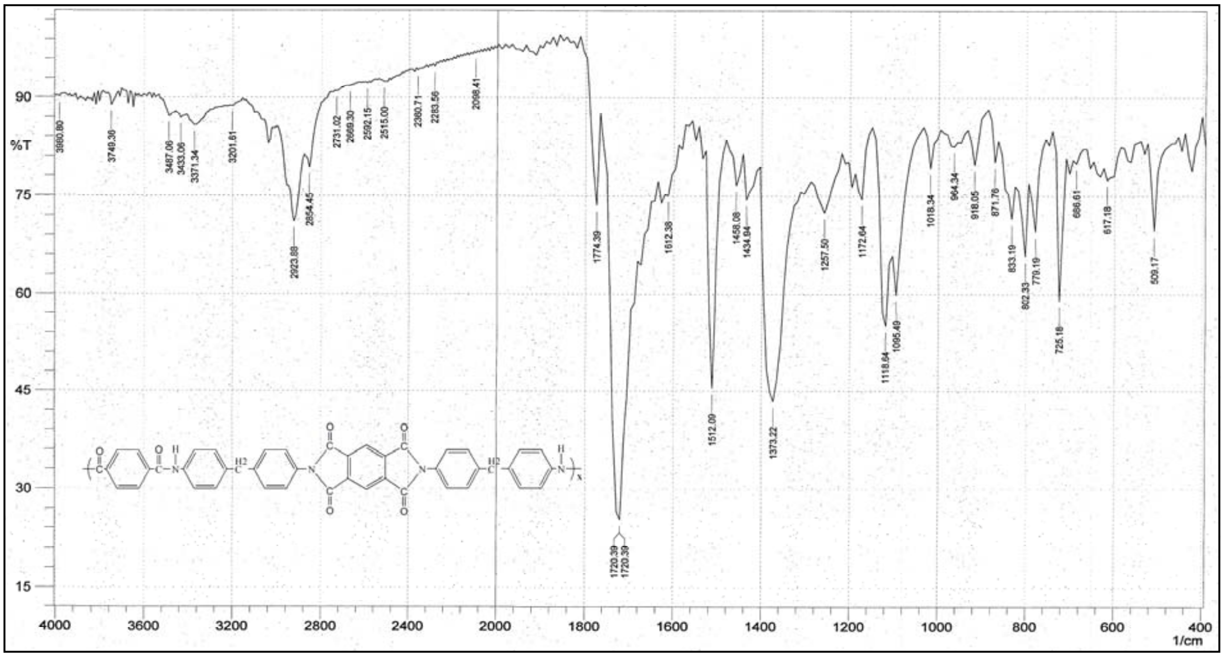


Figure No.(4): FTIR spectrum of (PAI1).

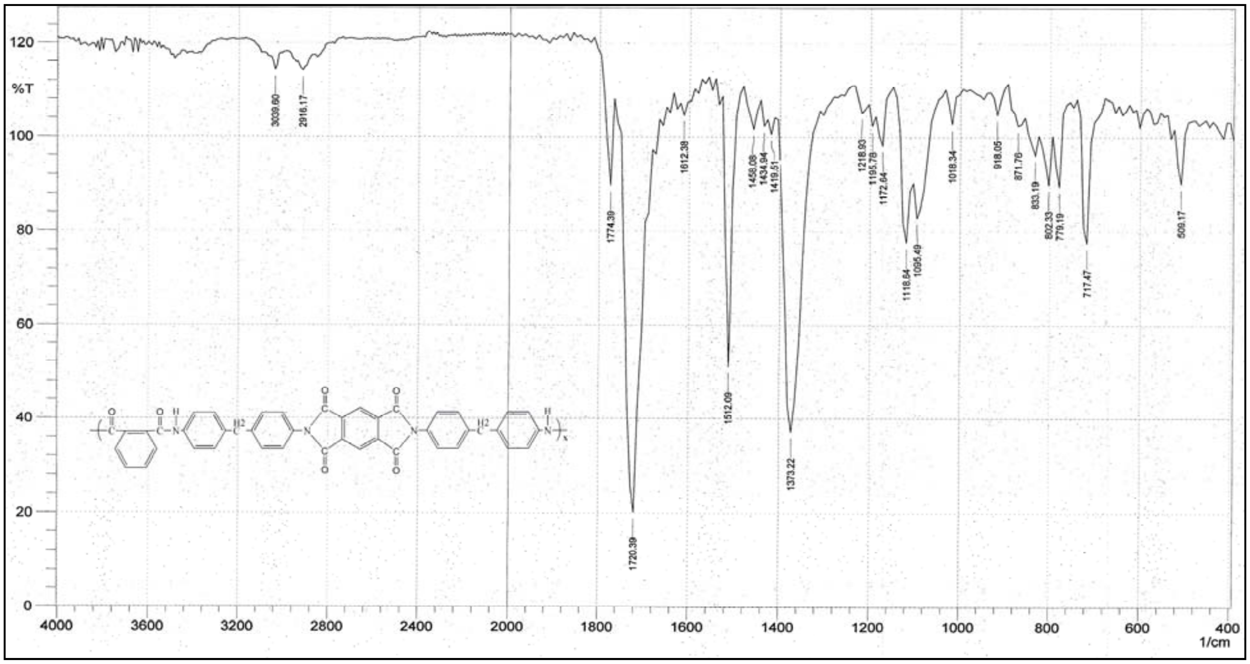


Figure No.(5) : FTIR Spectrum of (PAI2).

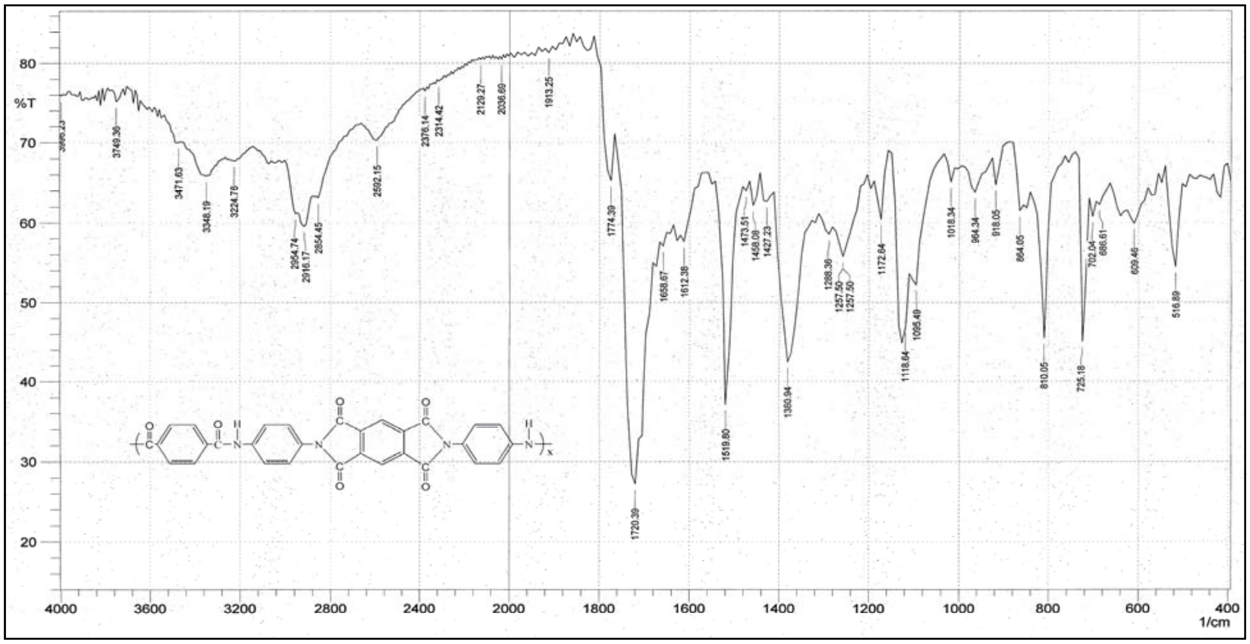


Figure No. (6): FTIR spectrum of (PAI3).

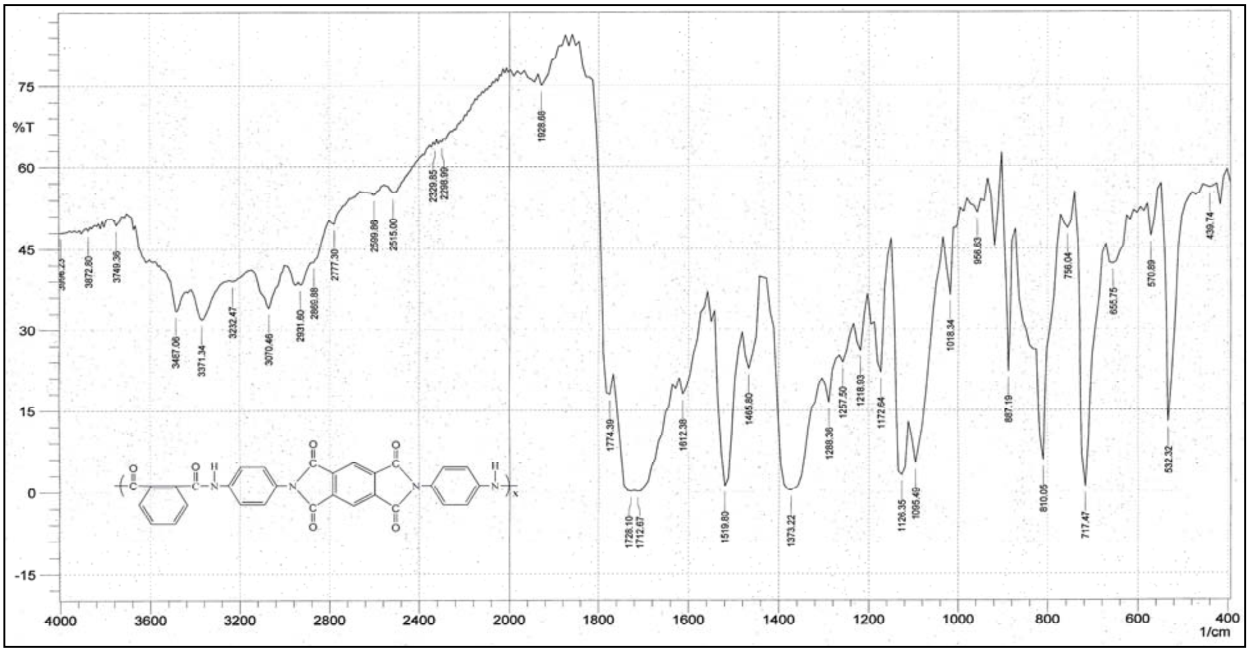


Figure No.(7) : FTIR spectrum of (PAI4).

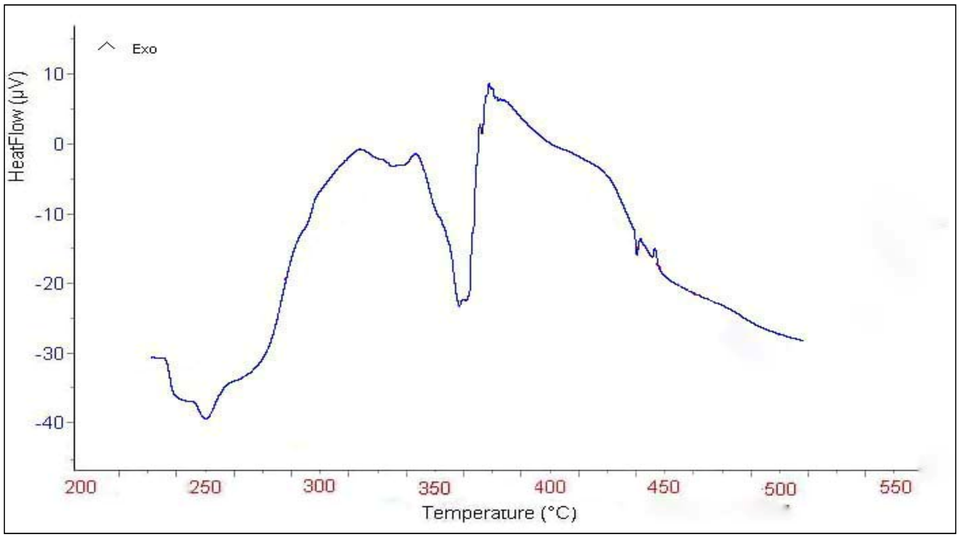


Figure No.(8): DSC curve of polymer (Pi-a1)

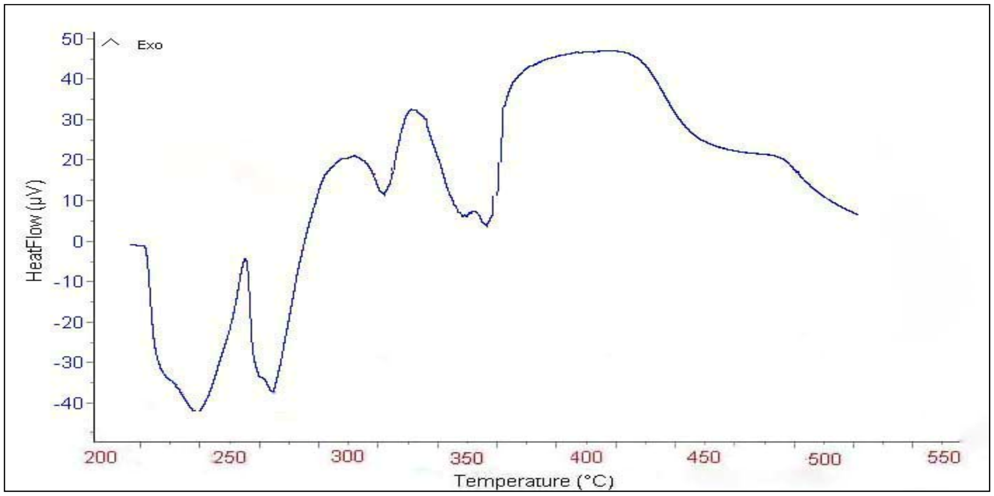


Figure No.(9): DSC curve of polymer (Pi-a2)

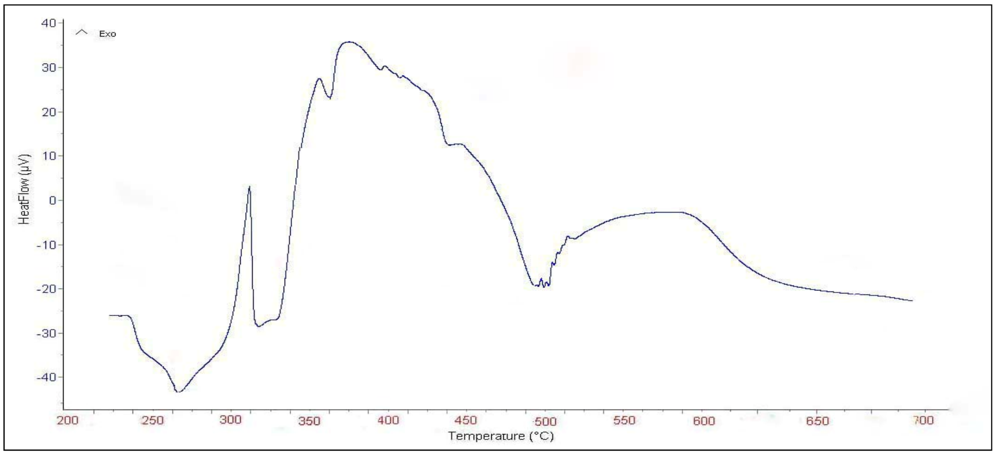


Figure No.(10) : DSC curve of polymer (Pi-a3)

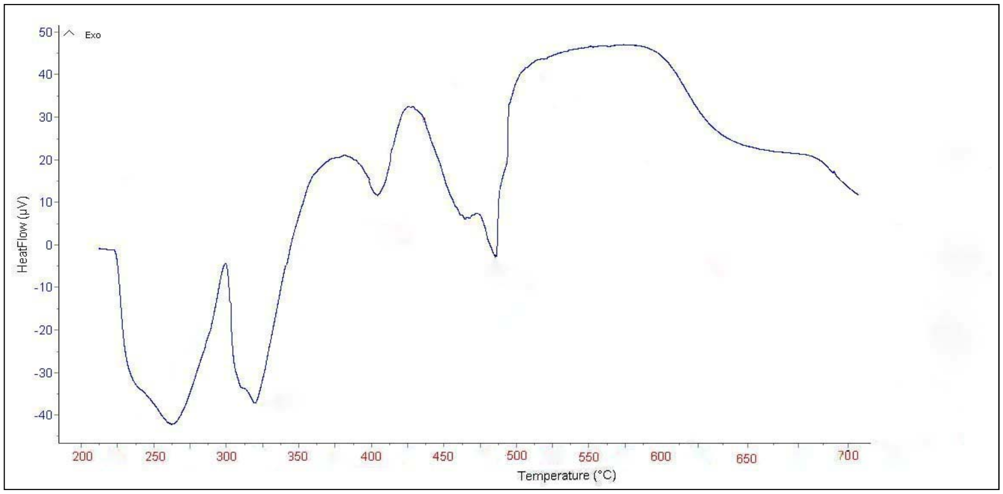
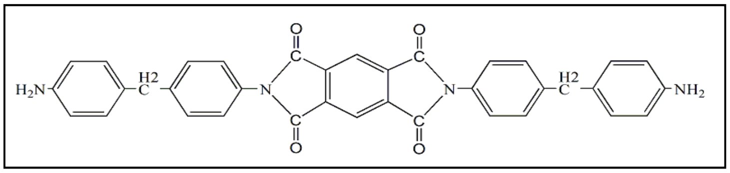
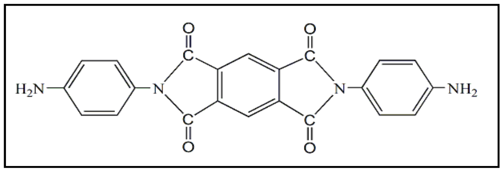


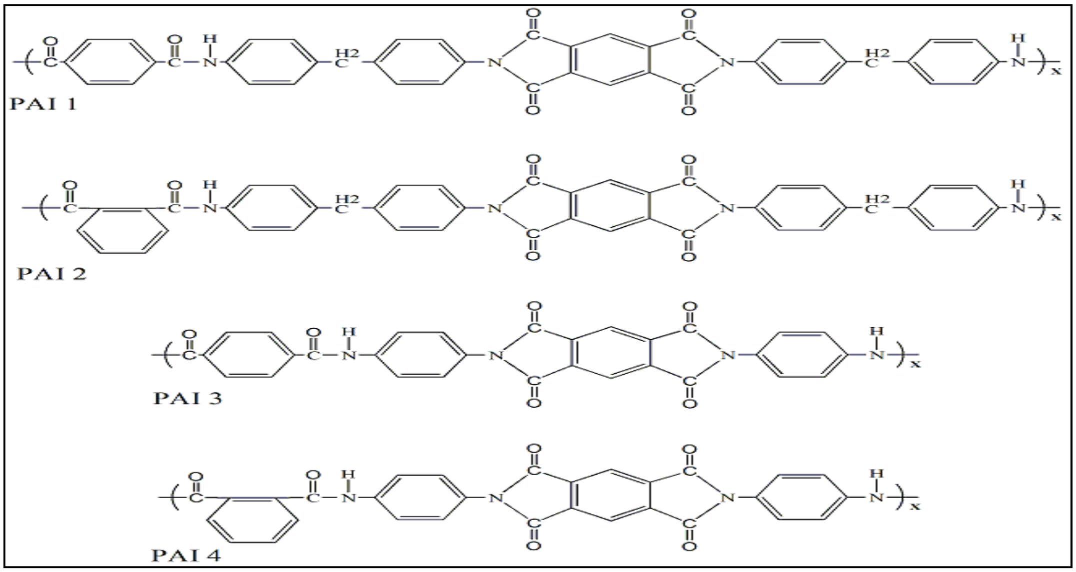
Figure N0.(11) : DSC curve of polymer (Pi-a4)



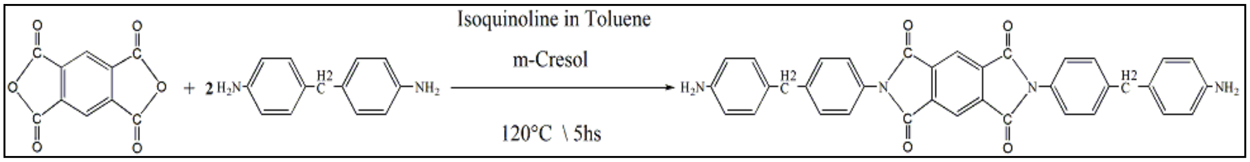
Scheme No.(1): Structure of M1.



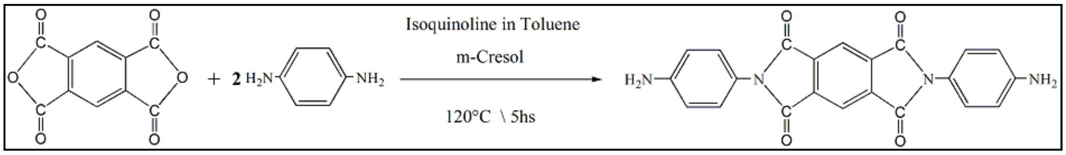
Scheme No.(2) : Structure of M2.



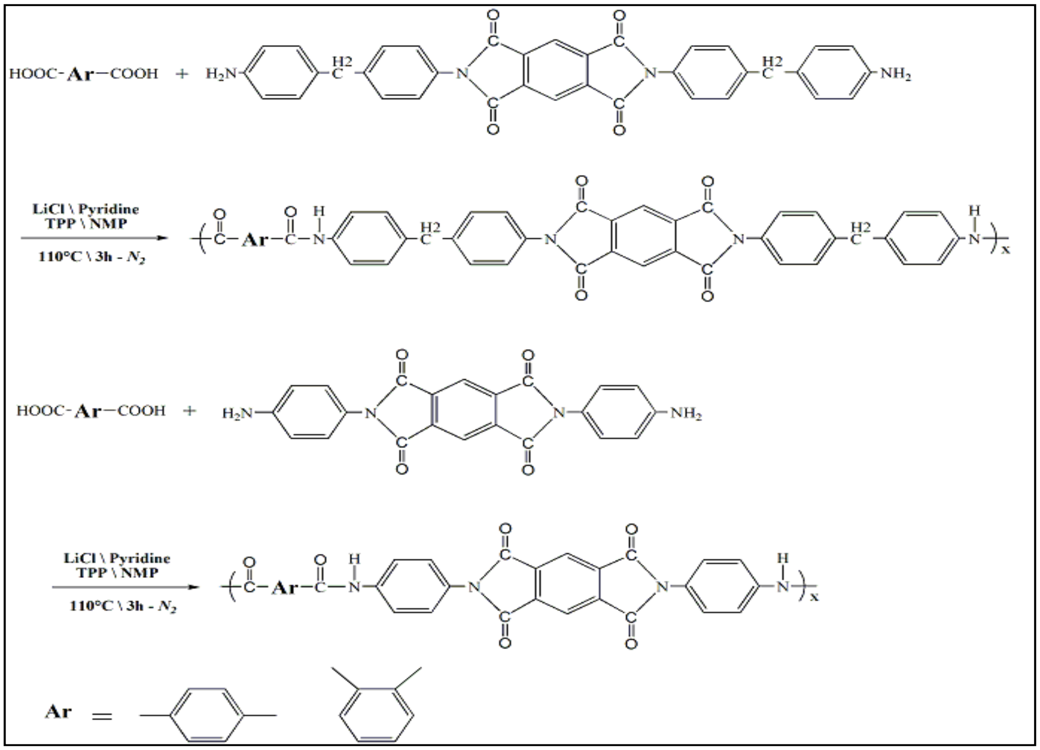
Scheme No.(3) : Repeating unit of PAI1- PAI4



Scheme No.(4) : Synthesis of M1.



Scheme No.(5) : Synthesis of(M2).



Scheme No.(6) : Synthesis of (PAI1- PAI4).

دراسة تخليق وتشخيص بولي (أميدات-إيميدات) أروماتية جديدة

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الخلاصة

حضرت بولي (أميدات-إيميدات) أروماتية جديدة مستقرة حراريا" من تفاعل بلمرة التكثيف المباشرة لحامض التيريفثالك وحامض الفثالك مع مونيمرين ثنائي الأمين مختلفين جديدين مشتقين من حامض 5,4,2,1-رباعي كاربوكسيليك بنزين ثنائي الانهيدرايد في وسط مؤلف من ثلاثي فينيل فوسفيت في ن-مثيل -2-بايروليدينون في محلول البيريدين مكون من كلوريد الكالسيوم الذائب. أنتج تفاعل البلمرة سلسلة من البولي(أميدات-إيميدات) الجديدة بناتج عال". المونيمرات الجديدة شخصت بتقانتي طيف الأشعة تحت الحمراء وطيف الرنين النووي المغناطيسي. وشخصت البوليمرات الناتجة عادة بتقانتي طيف الأشعة تحت الحمراء وطيف الرنين النووي المغناطيسي واختبارات الذوبانية. حققت الخواص الحرارية لهذه البوليمرات أيضا" باستعمال تقانة المسح المسعرين التفاضلي. وكشفت المعطيات التي ظهرت من التحليل الحراري كشفت بان هذه البوليمرات أظهرت استقرارية حرارية جيدة جدا. هذه البوليمرات تذوب بسهولة في المذيبات العضوية المتنوعة.

الكلمات المفتاحية :(بولي أميدات أروماتية , بولي إيميدات أروماتية , حامض 5,4,2,1-رباعي كاربوكسيليك بنزين ثنائي الانهيدرايد)