



Design and synthesis of novel homo and copolymerization based on 4-(N-maleimidylmethylbenzylidene)-4'-(N-citraconamic acid)-1,1'-biphenyl

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Abstract

The present work involved design and synthesis of six new polymaleimides, which contain three important moieties together namely maleimide ring, Schiff base and citraconamic acid. Synthesis of the new polymers was performed by many steps at the first step N-(4-acetophenyl) maleamic acid was prepared via reaction of 4-amino acetophenone with maleic anhydride. Dehydration of N-(4-acetophenyl) maleamic acid in the second step afforded N-(4-acetophenyl) maleimide and this in turn was presented in condensation reaction with benzidine in the third step generating 4-(N-maleimidylmethylbenzylidene)-4'-amino-1,1'-biphenyl, reaction of this compound with citraconic anhydride gave 4-(N-maleimidylmethylbenzylidene)-4'-(N-citraconamic acid)-1,1'-biphenyl. The last compound is the key compound and the new important monomer in this work which contains two vinylic bonds readily introduced in free radical homo and copolymerization producing the new homopolymer and copolymers. The presence of bulk Schiff base and citraconamic acid components beside co-polymerization exhibit the new polymers good fusibility and solubility leading to easier processability and wide applications.

Key words. copolymerization, polyimides, interchain forces, citraconamic acid.

1. Introduction

Polyimides represent durable and commercially important materials that exhibit high thermo-oxidative and thermal stabilities, high mechanical strength and good electrical properties [1-5]. Due to these properties interest in polyimides has increased and introduced in many applications like aerospace, medical, microelectronics, fuel cells and gas separation membranes [6-9]. However polyimides showed infusibility and low solution processability, due to high interchain forces of polymeric chains and this limited their applications. Thus the goal of polyimide chemists is preparation of polyimides that can be easily fabricated and this may be made either by incorporating suitable functional segments in polymers back bone or by copolymerization [10-12] producing modified polymers with better solubility and fusibility leading to easier processability and wider spectrum of applications. In this work we design and synthesize new monomer containing maleimide ring, citraconamic acid and Schiff base components together followed by free radical homo and copolymerization producing new polymaleimides

with modified properties.

2. Experimental Part

Chemicals were purchased from Merk, Fluka and BDH companies, FTIR spectra were recorded on SHIMADZU FTIR 8400 FT-IR spectrophotometer. ¹H-NMR and ¹³C-NMR spectra were recorded on nuclear magnetic resonance Bruker 400 MHz. Melting points were measured on Gallen Kamp capillary melting point apparatus while softening points were measured on thermal microscope Riechert thermover apparatus.

2.1. Preparation of N-(4-acetophenyl) maleamic acid (1)

The mentioned compound was prepared through reaction of equimolar amounts of 4-amino acetophenone and maleic anhydride in dry acetone as solvent with stirring for two hrs. at room temperature [13]. The resulted solid was filtered, dried followed by recrystallization from ethanol.

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2.2. Preparation of *N*-(4-acetophenyl) maleimide (2)

A mixture of (2.33 g, 0.01 mol) of compound (1) in (20 mL) acetic anhydride and sodium acetate (anhydrous) was refluxed for two hrs. with continuous stirring [14]. After completion of reflux time the mixture was poured onto ice D.W. with stirring and the formed solid was filtered, dried then recrystallized from acetone.

2.3. Preparation of 4-(*N*-maleimidylmethylbenzylidene)-4'-amino-1,1'-biphenyl (3)

A solution of (2.15 g, 0.01 mol) of compound (2) in (25 mL) of absolute ethanol with five drops of glacial acetic acid was added to solution of benzidine (1.84 g, 0.01 mol) dissolve in (15 mL) absolute ethanol then mixture was refluxed for 6 hrs. [15] after completion of reflux the formed solid was filtered, dried and recrystallized from ethanol.

2.4. Preparation of 4-(*N*-maleimidylmethylbenzylidene)-4'-(*N*-citraconamic acid)-1,1'-biphenyl (4)

A solution of (3.8 g, 0.01 mol) of compound (3) dissolved in (25 mL) of acetone was added as portions to solution of (1.12 g, 0.01 mol) of citraconic anhydride in (25 mL) acetone with stirring then the resulted mixture was stirred for 3 hrs. [13] the resulted solid was filtered, dried and recrystallized from ethanol.

2.5. homopolymerization of monomer 4-(*N*-maleimidylmethylbenzylidene)-4'-(*N*-citraconamic acid)-1,1'-biphenyl (5)

In polymerization bottle (0.002 mol, 0.98 g) of monomer compound (4) was placed then (15 mL) THF and (0.0002 g) of initiator (Azobisisobutyronitrile) AIBN were added then flushed with nitrogen gas, stoppered and heated at (75°C) for 3 hrs. [12] the resulted mixture was poured into methanol and the formed solid precipitate was filtered and dried.

2.6. Copolymerization of 4-(*N*-maleimidylmethylbenzylidene)-4'-(*N*-citraconamic acid)-1,1'-biphenyl with vinylic monomers (6-10)

Copolymer (6-10) were prepared by the same step used in preparation of polymer (5) except introducing both compound (4) and one of the following vinylic monomers together in polymerization process. The used vinylic monomers include acrylonitrile,

methacrylonitrile, methylacrylate, methylmethacrylate and vinyl acetate.

3. Result and Discussion

In this work we design and synthesize new maleimide monomer via introducing two bulk segments Schiff base and citraconamic acid in its structure then introduce it in homo and copolymerization producing new polymers have low fusibility and good solubility since these two points introducing of bulk segments in polymer backbone and copolymerization are excellent method for lowering interchain forces in polymer and this caused decreasing of softening points, increasing of solubility leading to good processability.

Synthesis of the desired polymers was accomplished by many steps which are shown in Scheme (1) and (2).

As indicated in scheme (1) the first step involved synthesis of compound (1) via reaction between 4-amino acetophenone and maleic anhydride. Compound (1) was dehydrated in the second step producing compound (2) *N*-(4-acetophenyl) maleimide. We used acetic anhydride in presence of anhydrous sodium acetate as dehydrating agent which caused elimination of water molecule and ring-closure giving the imide compound (2).

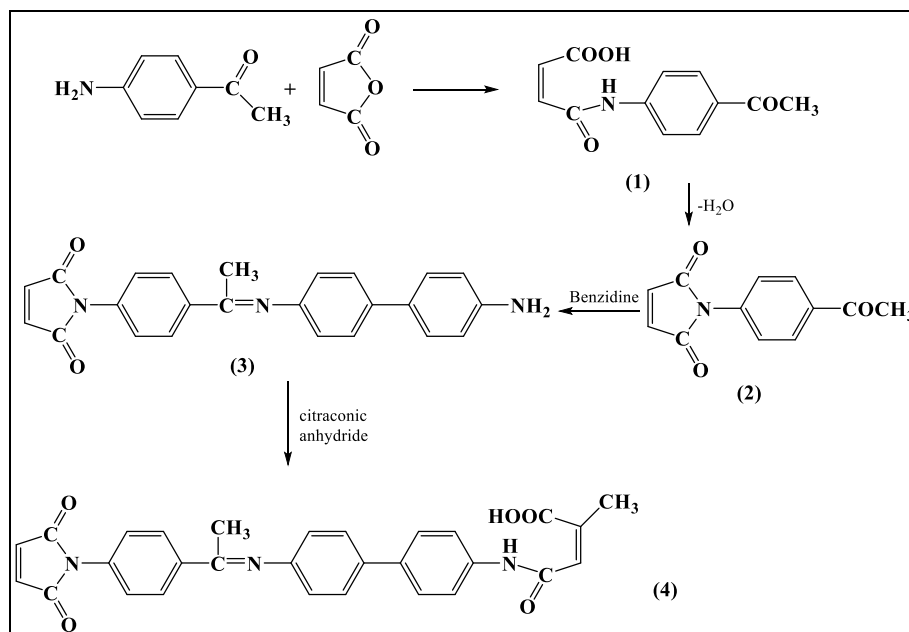
The presence of aceto group in compound (2) gave the chance for introducing in condensation reaction with benzidine affording Schiff base compound (3) which inturn contain amino group ready for introducing in reaction with citraconic anhydride producing compound (4) 4-(*N*-maleimidylmethylbenzylidene)-4'-(*N*-citraconamic acid)-1,1'-biphenyl.

Physical properties of compound (1-4) are shown in table (1).

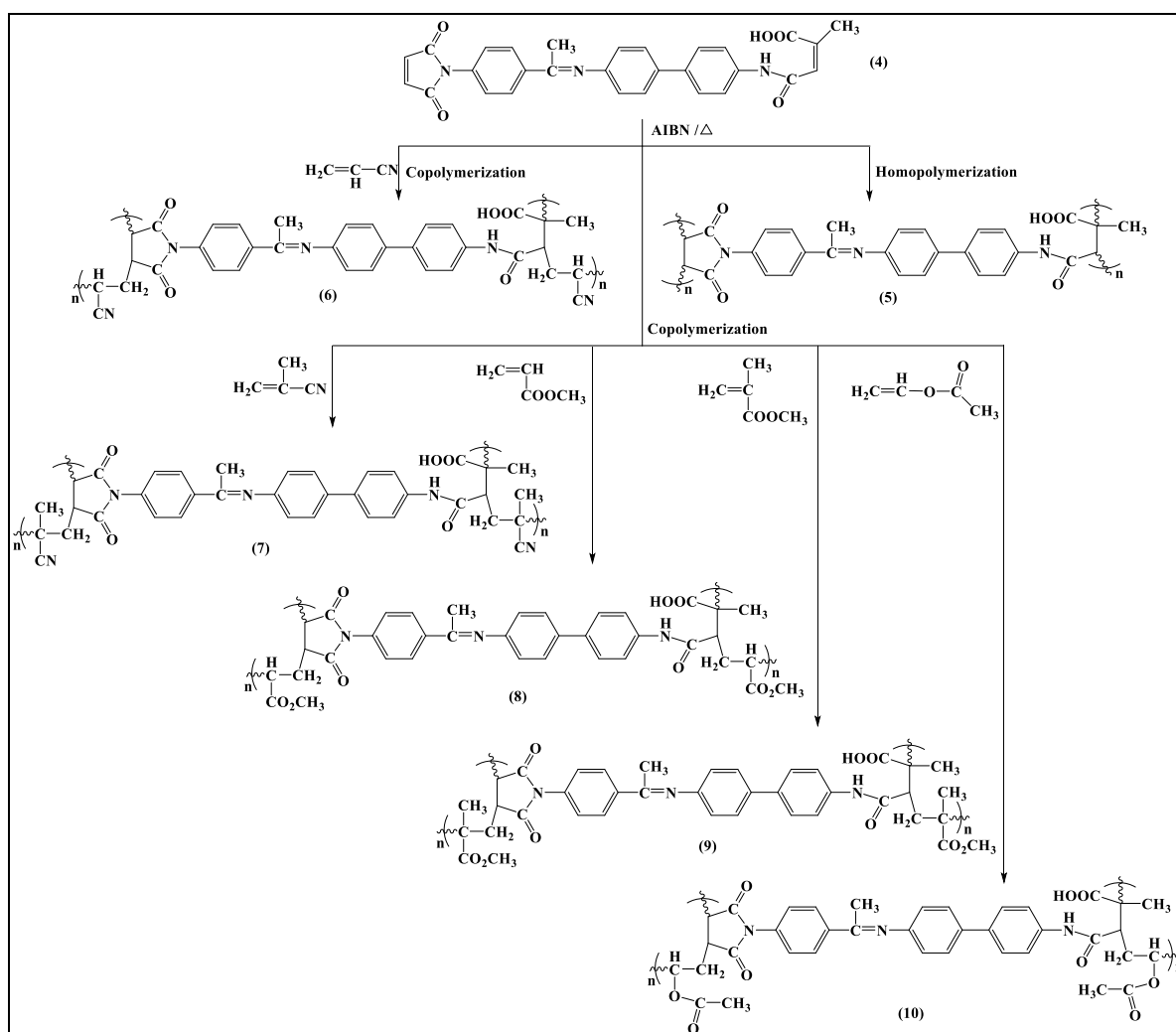
FTIR spectrum of compound (1) showed absorption bands at (3459, 3417, 3269) cm^{-1} due to $\nu(\text{N-H})$ and $\nu(\text{O-H})$. Further bands at (1710, 1677, 1633) cm^{-1} due to $\nu(\text{C=O})$ carboxyl, $\nu(\text{C=O})$ ketone and $\nu(\text{C=O})$ amide [16].

FTIR spectrum of compound (2) showed appearance of shoulder band at (1778) cm^{-1} and other at (1714) cm^{-1} owing to $\nu(\text{C=O})$ imide.

Besides the spectrum showed disappearance of absorption bands belong to $\nu(\text{C=O})$ amide and $\nu(\text{O-H})$, $\nu(\text{N-H})$ proving success of imide formation [16].



Scheme (1) synthetic route of compounds (1-4)



Scheme (2) synthetic route of polymers (5-10)

FTIR spectrum of compound (3) presented appearance of two bands due to asym. and sym. $\nu(\text{NH}_2)$ at $(3359) \text{ cm}^{-1}$ and $(3201) \text{ cm}^{-1}$ beside disappearance of $\nu(\text{C}=\text{O})$ ketone band and appearance of $\nu(\text{C}=\text{N})$ absorption band at $(1658) \text{ cm}^{-1}$ proving success of Schiff base compound (3) formation.

$^1\text{H-NMR}$ spectrum of compound (3) displayed signals at $(\sigma=2.63, 5.18, 6.61) \text{ ppm}$ and at $(\sigma=7.21, 8.05) \text{ ppm}$ which are belong to methyl protons, (NH_2) protons, vinylic protons and aromatic protons respectively [17].

FTIR spectrum of compound (4) displayed bands at $(3307) \text{ cm}^{-1}$ and $(3203) \text{ cm}^{-1}$ owing to both $\nu(\text{O-H})$ carboxyl and $\nu(\text{N-H})$ amide. Other bands seemed at $(1724), (1675)$ and $(1627) \text{ cm}^{-1}$ which are due to $\nu(\text{C}=\text{O})$ imide, $\nu(\text{C}=\text{O})$ carboxyl and $\nu(\text{C}=\text{O})$ amide. All details of FTIR spectral data of compounds (1-4) are listed in Table (4).

Compound (4) is the important newly design and synthesized monomer in this work that contain two vinylic bonds ready for polymerization beside containing three important segments maleimide ring, Schiff base and citraconamic acid and every one of them exhibit the polymers desired properties.

As indicated in scheme (2) compound (4) was introduced in homopolymerization producing homopolymer (5) and in copolymerization with five vinylic monomers producing copolymers (6-10). By copolymerization new repeating units were introduced in polymer backbone and this exhibit the polymer flexibility, decreasing softening points and increasing solubility [12].

The new polymers was afforded in high conversion ratio (74-81)% as orange to reddish brown solids with low softening points and some of them are gummy. They showed good solubility in many solvents including THF, DMF, DMSO and dioxane.

Physical properties of polymers (5-10) are shown in Table (2) while their solubility data are listed in Table (3).

FTIR spectrum of polymers (5-10) displayed bands at $(3382-3448) \text{ cm}^{-1}$ and $(3203-3317) \text{ cm}^{-1}$ owing to $\nu(\text{O-H})$ carboxyl and $\nu(\text{N-H})$ amide [16]. Other absorption bands seemed at $(1760-1772) \text{ cm}^{-1}$, $(1700-1733) \text{ cm}^{-1}$, $(1670-1700) \text{ cm}^{-1}$, $(1627-1689) \text{ cm}^{-1}$, $(1600-1630) \text{ cm}^{-1}$ and $(1348-1396) \text{ cm}^{-1}$ which are due to asym. $\nu(\text{C}=\text{O})$ imide, sym. $\nu(\text{C}=\text{O})$ imide, $\nu(\text{C}=\text{O})$ carboxyl, $\nu(\text{C}=\text{O})$ amide, $\nu(\text{C}=\text{N})$ and $\nu(\text{C}=\text{N})$ imide respectively [16].

FTIR spectrum of copolymers (6) and (7) showed absorption bands at $(2237-2244) \text{ cm}^{-1}$ due to $\nu(\text{C}\equiv\text{N})$ while FTIR spectrum of copolymers (8-10) exposed bands at $(1718-1733) \text{ cm}^{-1}$, $(1242) \text{ cm}^{-1}$ and $(1184-1199) \text{ cm}^{-1}$ due to $\nu(\text{C}=\text{O})$ ester and $\nu(\text{C}-\text{O})$ ester.

FTIR spectral data of polymers (5-10) are shown in Table (5).

$^1\text{H-NMR}$ spectrum of polymer (6) showed signals at $(\sigma=1.29-1.96) \text{ ppm}$ belong to $(-\text{CH}_2-)$ protons and $(-\text{CH}-\text{CH}-)$, $(-\text{CH}-)$ protons, in imide and acid moieties. Other signals appeared at $(\sigma=2.1)$, $(\sigma=2.73-2.76)$, $(\sigma=3.74-3.95)$, $(\sigma=6.91-8.04)$, $(\sigma=8.2)$ and $(\sigma=10.4) \text{ ppm}$ which are belong to (CH_3) citracon, (CH_3) imine, $(\text{NC}-\text{CH})$ protons, aromatic protons, (NH) amide and (OH) acid protons respectively [17].

$^{13}\text{C-NMR}$ spectrum of polymer (6) showed signals at $(\sigma=14.11-36.61) \text{ ppm}$ belong to $(-\text{CH}_2-)$, imide $(-\text{CH}-\text{CH}-)$ carbons (CHCONH) carbon, (CH_3) citracon and (CH_3) imine carbons. Other signals at $(\sigma=66.88-66.99) \text{ ppm}$, $(\sigma=67.03-67.30) \text{ ppm}$, $(\sigma=75.58-75.74) \text{ ppm}$, $(\sigma=119.32-138.07) \text{ ppm}$, $(\sigma=142.76) \text{ ppm}$, $(\sigma=162.62) \text{ ppm}$ and $(\sigma=170.19) \text{ ppm}$ which are belong to $(\text{NC}-\text{CH})$, $(-\text{C}-\text{COOH})$, $(\text{C}\equiv\text{N})$, aromatic carbons, $(\text{C}=\text{N})$, $(\text{C}=\text{O})$ amide and acid and $(\text{C}=\text{O})$ imide carbons respectively.

$^1\text{H-NMR}$ spectrum of polymer (7) showed signals at $(\sigma=1.29-1.98) \text{ ppm}$ belong to $(-\text{CH}_2-)$, $(-\text{CH}-\text{CH}-)$ imide and $(-\text{CH}-)$ acid protons. Other signals appeared at $(\sigma=2.11-2.2) \text{ ppm}$, $(\sigma=2.73) \text{ ppm}$, $(\sigma=6.92-8.05) \text{ ppm}$ and $(\sigma=10.38) \text{ ppm}$ which belong to three methyl groups, (CH_3) imine, aromatic protons, (NH) amide proton and (OH) acid proton.

$^{13}\text{C-NMR}$ spectrum of polymer (7) showed signals at $(\sigma=20.50-36.21) \text{ ppm}$ belong to $(-\text{CH}_2-)$, four methyl groups carbons, $(-\text{CH}-\text{CH}-)$ imide and $(-\text{CH}-)$ in amic acid carbons. Other signals appeared at $(\sigma=51.83) \text{ ppm}$, $(\sigma=56-58) \text{ ppm}$, $(\sigma=66) \text{ ppm}$, $(\sigma=113.32-138.07) \text{ ppm}$, $(\sigma=142.70) \text{ ppm}$, $(\sigma=162.60) \text{ ppm}$, $(\sigma=170.19) \text{ ppm}$ and $(\sigma=176.15) \text{ ppm}$ which are belong to $(-\text{C}-\text{C}\equiv\text{N})$, $(-\text{C}-\text{COOH})$, $(\text{C}\equiv\text{N})$, aromatic carbons, $(\text{C}=\text{N})$, $(\text{C}=\text{O})$ amide, $(\text{C}=\text{O})$ carboxyl and $(\text{C}=\text{O})$ imide carbons respectively [17].

$^1\text{H-NMR}$ spectrum of polymer (8) showed signals at $(\sigma=1.28-2.18) \text{ ppm}$ belong to $(-\text{CH}_2-\text{CH}-)$ protons, $(-\text{CH}-\text{CH}-)$ imide, $(-\text{CH}-)$ amic acid and (CH_3) citracon protons. Other signals appeared at $(\sigma=2.65) \text{ ppm}$, $(\sigma=3.69) \text{ ppm}$, $(\sigma=7.63-7382) \text{ ppm}$, $(\sigma=8.21) \text{ ppm}$ and $(\sigma=10.39) \text{ ppm}$ which belong to (CH_3) imine, (OCH_3) , aromatic protons, (NH) amide and (OH) carboxyl protons [16].

Table (1) physical properties of compounds (1-4)

Comp. No.	Compound structure	Color	Melting point °C	Yield %	Recrystallization solvent
1		Yellow	201-203	94	Ethanol
2		Dark red	279-281	96	Acetone
3		Brown	77-78	75	Ethanol
4		Brown	178-180	88	Ethanol

Table (2) Physical properties of polymers (5-10)

Poly. No.	polymer structure	Color	Softening point °C	Conv. Ratio	Purification
5		Brown	203-215	79	
6		Reddish brown	Gummy	81	
7		Orange	110-125	74	
8		Brown	Gummy	77	By dissolving in THF then precipitation by methanol
9		Brown	80-97	80	
10		Pale brown	Gummy	85	

Table (3) Solubility data of polymers (5-10)

Poly. No.	THF	DMF	DMSO	Acetone	MeOH	EtOH	Dioxane	CHCl ₃	n-hexane	Petr. ether	Et ₂ O
5	s	s	s	ins	ins	ins	s	s	ins	ins	ins
6	s	s	s	ins	ins	ins	s	s	ins	ins	ins
7	s	s	s	ins	ins	ins	s	s	ins	ins	ins
8	s	s	s	ins	ins	ins	s	s	ins	ins	ins
9	s	s	s	ins	ins	ins	s	s	ins	ins	ins
10	s	s	s	ins	ins	ins	s	s	ins	ins	ins

(s= soluble, ins= insoluble)

Table (4) FTIR spectral data (cm⁻¹) of compounds (1-4)

Comp. No.	$\nu(\text{O-H})$ $\nu(\text{N-H})$	$\nu(\text{C-H})$ Arom.	$\nu(\text{C-H})$ Alipha.	$\nu(\text{C=O})$ Acid	$\nu(\text{C=O})$ Imide	$\nu(\text{C=O})$ Amide	$\nu(\text{C=C})$	$\nu(\text{C-N})$ Imide	$\nu(\text{C=N})$	Other
1	3459 3417 3269	3087	2920 2870	1710	-	1633	1537	-	-	$\nu(\text{C=O})$ Ketone 1677
2	-	3053	2916 2880	-	1778 1714	-	1598	1379	-	$\nu(\text{C=O})$ Ketone 1679
3	-	3029	2927 2867	-	1778 1710	-	1606	1384	1658	$\nu(\text{NH}_2)$ 3359 3201
4	3307 3203	3060	2968 2880	1675	1724	1627	1527	1330	1569	-

Table (4) FTIR spectral data (cm⁻¹) of compounds (1-4)

Comp. No.	$\nu(\text{O-H})$ $\nu(\text{N-H})$	$\nu(\text{C-H})$ Arom.	$\nu(\text{C-H})$ Alipha.	$\nu(\text{C=O})$ Acid	$\nu(\text{C=O})$ Imide	$\nu(\text{C=O})$ Amide	$\nu(\text{C=C})$	$\nu(\text{C-N})$ Imide	$\nu(\text{C=N})$	Other
1	3459 3417 3269	3087	2920 2870	1710	-	1633	1537	-	-	$\nu(\text{C=O})$ Ketone 1677
2	-	3053	2916 2880	-	1778 1714	-	1598	1379	-	$\nu(\text{C=O})$ Ketone 1679
3	-	3029	2927 2867	-	1778 1710	-	1606	1384	1658	$\nu(\text{NH}_2)$ 3359 3201
4	3307 3203	3060	2968 2880	1675	1724	1627	1527	1330	1569	-

¹³C-NMR spectrum of polymer (8) showed signals at (σ =20.51-34.26) ppm belong to (-CH₂-), (-CH-CH-) imide, (-CH-) amic acid, (CH₃) citracon and (CH₃) imine carbons. Other signals appeared at (σ =51.31) ppm, (σ =51.51) ppm, (σ =66) ppm, (σ =113.0-138.07) ppm (σ =142.72) ppm, (σ =162.61) ppm, (σ =170.18) ppm and (σ =174.49) ppm which are belong to (-C-COOH), (-CH-CO), (OCH₃), aromatic carbons, (C=O) amide and carboxyl, (C=N), (C=O) imide and (C=O) ester carbon respectively.

¹H-NMR spectrum of polymer (9) showed signals at (σ =0.84-2.17) ppm belong to (-CH₂-) protons, (-CH-CH-) imide, (-CH-) amic acid and protons of three methyl groups [16]. Other signals appeared at (σ =2.74) ppm, (σ =3.76) ppm, (σ =7.71-8.1) ppm,

(σ =8.25) ppm and (σ =10.4) ppm which belong to (CH₃) imine, (OCH₃), aromatic protons, (NH) amide and (OH) carboxyl protons.

¹³C-NMR spectrum of polymer (9) showed signals at (σ =18.42-29.76) ppm belong to (-CH₂-), (-CH-CH-) imide, (-CH-) amic acid carbons and carbons of four methyl groups. Other signals appeared at (σ =50.54-53.73) ppm, (σ =66.72) ppm, (σ =113.31-142.69) ppm, (σ =162.60), (σ =170.18) ppm, (σ =176.19) ppm, (σ =176.51) ppm and (σ =177.16-177.37) ppm which are belong to (-C-COOH), (-CH-CO), (OCH₃), (C=N), aromatic carbons, (C=O) amide, (C=O) acid, (C=O) imide and (C=O) ester carbon [17].

Table (5) FTIR spectral data (cm⁻¹) of polymers (5-10)

Poly. No.	$\nu(\text{O-H})$ $\nu(\text{N-H})$	$\nu(\text{C-H})$ Arom.	$\nu(\text{C-H})$ Aliphatic	$\nu(\text{C=O})$ Imide	$\nu(\text{C=O})$ Carboxyl	$\nu(\text{C=O})$ Amide	$\nu(\text{C=N})$	$\nu(\text{C=C})$	$\nu(\text{C-N})$ Imide	Others
5	3448 3309 3205	3062	2968 2877	1760 1726	1670	1627	1569	1531	1384	-
6	3435 3317 3215	3060	2974 2881	1760 1716	1677	1633	1604	1529	1392	$\nu(\text{C}\equiv\text{N})$ 2244
7	3446 3311 3203	3064	2981 2885	1770 1712	1681	1631	1600	1525	1396	$\nu(\text{C}\equiv\text{N})$ 2237
8	3382 3313	3024	2954 2883	1733	1685	1652	1630	1602	1382	$\nu(\text{C=O})$ Ester 1733 $\nu(\text{C-O})$ Ester 1199 $\nu(\text{C=O})$ Ester 1731
9	3420 3313	3020	2995 2880	1700	1700	1643	1602	1535	1394	$\nu(\text{C-O})$ Ester 1242 1193 $\nu(\text{C=O})$ Ester 1718
10	3448 3417	3024	2923 2904	1772 1718	1689	1689	1600	1560	1348	$\nu(\text{C-O})$ Ester 1242 1184

4. Conclusions

- The synthesis of polyimides with schiff base group in polymer backbone leads to improve the properties of polymers and give it a more flexibility and processability.
- The new synthesized polymers have good solubility in many organic solvents.
- The second strategy for improve the properties of polyimides is presented the synthesized monomer in copolymerization with vinylic monomers which introduced new functional groups in polymers for new properties where improved the physical properties of polymers.
- Carboxylic acid group in polymer give a chance introduce polymers in new applications after make improve on this group.

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6. References

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