



Synthesis and Polymerization of Several New Maleimides Linked to Schiff Bases and Their Copolymers with Acrylonitrile

Ahلام Marouf Al-Azzawi, Esraa Faiq*

Department of Chemistry, College of Science, University of Baghdad, Baghdad, Iraq.

Abstract

A series of new maleimide monomers substituted with Schiff base moieties were synthesized via acid-catalyzed condensation of 3-(N-maleimidyl)phenyl hydrazide with aromatic aldehydes and ketones. The newly synthesized monomers were introduced in free radical chain growth homopolymerization producing five new polymaleimides with pendent Schiff base moieties. The new monomers were introduced also in free radical chain growth copolymerization with acrylonitrile producing new five copolymaleimides containing pendent Schiff base moieties. The new homopolymers and copolymers are of great importance since incorporation of bulky Schiff base moieties in their repeating units exhibit them better solubility and processing properties which made them suitable for a variety of important applications.

Keywords: Homopolymerization, Pendent Schiff base, Copolymaleimides.

تحضير وبلمره عدد من المالي ايميدات الجديدة المرتبطة بقواعد شيف وبوليمراتها المشتركة مع الأكريلونايتريل

أحلام معروف العزاوي، اسراء فائق*

قسم الكيمياء، كلية العلوم، جامعة بغداد، بغداد، العراق.

الخلاصة

تضمن البحث تحضير عدد من مونوميرات المالي ايميدات المعوضة بقواعد شيف وذلك من خلال تكاثف المركب 3-(N-مالي ايميديل) فنييل هيدرازيد مع الديهايدات وكيثونات اروماتية. تم ادخال المونوميرات المحضرة في تفاعل بلمره متسلسلة جذرية مما أسفر عن انتاج خمسة بولي مالي ايميدات جديدة تحتوي على مجاميع متدللية من قواعد شيف.

كذلك تم ادخال مونوميرات المالي ايميدات المحضرة في تفاعل بلمره مشتركة متسلسلة جذرية مع الأكريلونايتريل مما أسفر عن تكوين خمسة من بولي ايميدات المشتركة الجديدة والتي تحتوي على مجاميع متدللية من قواعد شيف.

تعتبر كل من البوليمرات الذاتية والمشاركة المحضرة في هذا البحث غاية في الأهمية نظراً لاحتواء وحداتها المتكررة على مكونة متدللية كبيرة (قاعدة شيف) والتي تمنح البوليمرات قابلية ذوبان أفضل وصفات تصنيعية أفضل مما يجعلها مناسبة للاستخدام في العديد من التطبيقات المهمة.

*Email: esraa faiq65@g-mail.com

Introduction

Polymaleimides represent a class of rigid polymers having excellent thermal stability, high mechanical and electrical properties beside good chemical resistance[1-3]. In spite of all these properties, poor solubility and processability limit the applications of polyimides[4-6].

Many efforts have been made to solve these problems since developing these properties can be an important approach to their use in many new fields [7-13]. Thus in the present work we try to improve polymaleimides properties by two ways the first one involved introducing of bulk Schiff base moieties as pendant groups in polymeric chains of polymaleimides, while the second way involved introducing of maleimides substituted with Schiff bases into copolymerization with acrylonitrile since copolymerization is the most efficient method for modifying polymer properties [14-17].

The present paper describes the synthesis of new maleimides linking to Schiff bases moieties followed by their homopolymerization and copolymerization producing both new polymaleimides and copolyimides with enhanced properties which may serve different applications.

Experimental

All employed chemicals were purchased from Merck, Fluka and BDH companies and used without further purification. FTIR spectra were recorded on Shimadzu FTIR-8400 Fourier Transform Infrared spectrophotometer as KBr disc. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra were run on a Bruker ultra shield 300MHz NMR spectrometer using DMSO- d_6 as solvent and tetramethylsilane as internal standard. Uncorrected melting points were determined on Thomas Hoover apparatus. Softening points were determined on thermal microscope Riechert thermover.

1. Preparation of ethyl-3-(N-maleimidyl)benzoate (1)

Compound (1) was prepared according to literature procedure[18] via reaction of (0.01 mol, 2.17g) of 3-(N-maleimidyl)benzoic acid with excess ethanol (30mL) in presence of H_2SO_4 catalyst under reflux for 8hrs. The resulted product was recrystallized from acetone producing white crystals in (60%) yield and melting point = 50-52°C.

2. Preparation of 3-(N-maleimidyl)phenyl hydrazide (2)

Compound (2) was prepared according to literature procedure[18] via refluxing, mixture of (0.01 mol, 2.45g) of compound (1) (0.02 mol, 1g) of hydrazine hydrate 98% for 4hrs followed by addition of (15mL) of ethanol then reflux was continued for 8hrs. The obtained solid was filtered, dried and recrystallized from ethanol producing off white crystals in (65%) yield with m.p = 66-68°C.

3. Preparation of Schiff bases (3-7)

Synthesis of Schiff bases (3-7) was performed via refluxing the mixture of (0.01 mol, 2.31g) of compound (2) with (0.01 mol) of aldehyde or ketone dissolved in (25 mL) of absolute ethanol with few drops of glacial acetic acid for 6hrs. with stirring [19]. The resulted solid was filtered, dried then purified by recrystallization from a suitable solvent.

4. Preparation of homo polymers (8-12)

In dry polymerization bottle (0.005mol) of Schiff base (3-7) was dissolved in (10mL) of THF then (0.002g) of AIBN (Azo bis iso butyronitrile) initiator was added then the bottle was flushed with nitrogen gas and stoppered [20]. The mixture was heated at 80 °C for 3hrs before pouring into (15mL) of methanol and the obtained solid polymer was filtered then purified by dissolving in THF followed by reprecipitation from methanol.

5. Preparation of copolymers (13-17)

Synthesis of copolymers (13-17) was performed by following the same procedure steps used in preparation of homopolymers (8-12) except using of equal weights of both Schiff base (3-7) and acrylonitrile which were dissolved together in THF solvent in presence of AIBN initiator. The obtained copolymer was filtered and purified by dissolving in THF followed by precipitation from methanol.

Results and discussion

Many attempts have been made to modify polymaleimides processing and solubility properties. One of these methods involved introducing of bulky units into polymeric chains[17] to affect their properties, thus in the present work we performed structure modifications by introducing bulky pendant (Schiff base) groups on polymaleimide backbone through preparation of new maleimide monomers substituted with Schiff base moieties then introduce them in homopolymerization. Besides it was reported [17] that using of meta-substituted instead of para-substituted phenyl monomers was very efficient method for reducing geometrical symmetry of repeating units in polymeric chains

leading to decrease interchain forces and this inturn enhanced solubility and decreased softening points of the resulted polymers and this is exactly what we made in this work since bulky Schiff base moieties in the prepared monomers are present at meta position of phenyl ring.

Synthesis of the new polymers was performed by many steps which are shown in Scheme 1. In the first step 3-(N-maleimidyl)benzoic acid which was prepared by reaction of 3-amino benzoic acid with maleic anhydride in glacial acetic acid was used as starting material that introduced in acid-catalyzed esterification reaction with ethanol producing compound 1 (1) which inturn was introduced in reaction with hydrazine hydrate producing hydrazide (2). Introducing of compound (2) in acid catalyzed condensation reaction [19] with different aldehydes and ketones in the third step produced several new maleimide monomers (3-7) substituted with Schiff base moieties at meta-position. Subsequently the newly synthesized monomer (3-7) were introduced in free radical chain growth homopolymerization [20] using THF as solvent and AIBN (Azo bis iso butyronitrile) as initiator to produce the target new polymers (8-12) polymaleimides with pendent bulk Schiff base groups. Physical properties of monomers (3-7) and homopolymers (8-12) are shown in Tables-1 and 2.

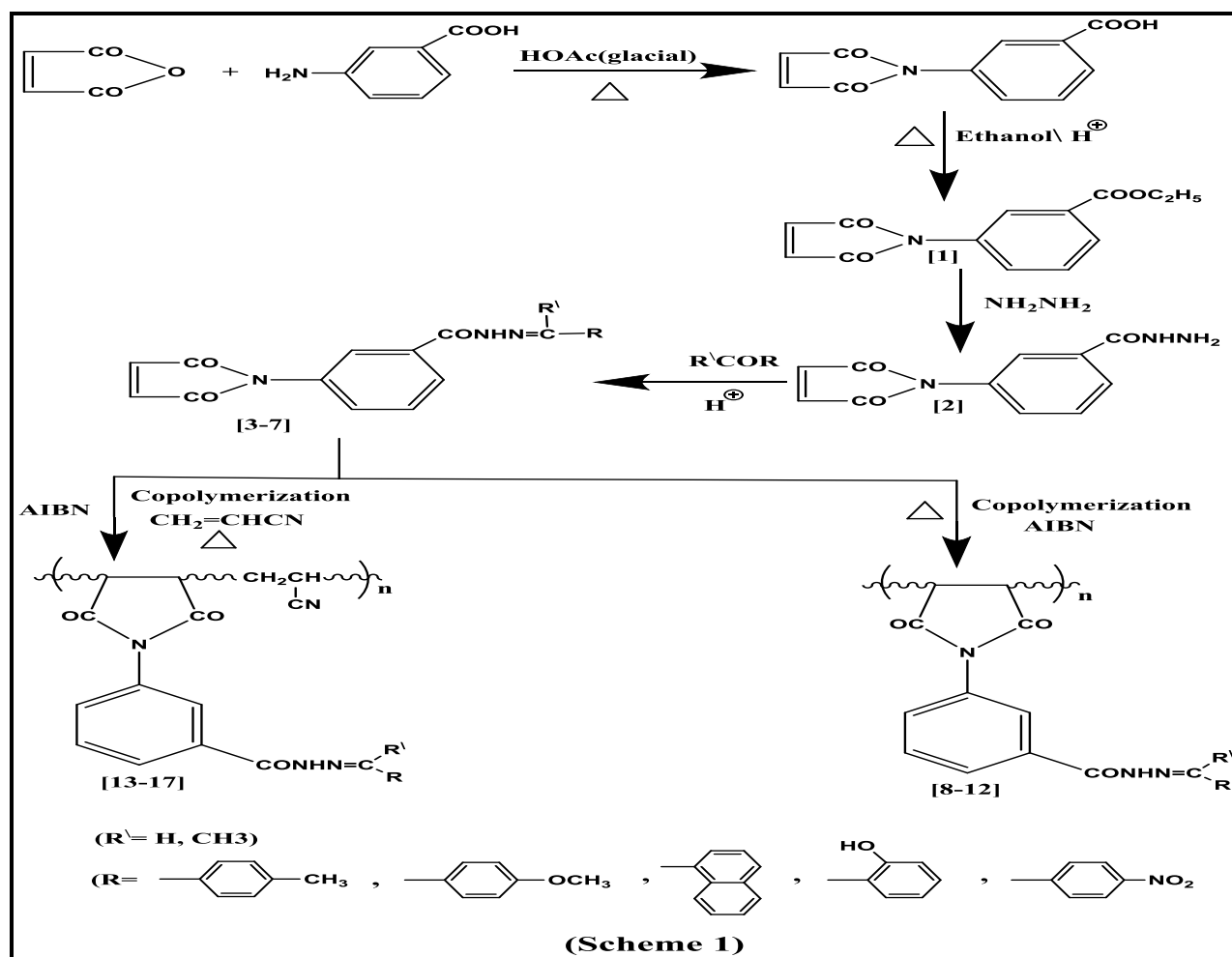


Table 1- Physical properties of Schiff bases (3-7)

Comp. No.	Compound structure	color	Yield%	Melting point C°	Recrystallization solvent
3		yellow	77	133-134	Acetone
4		Dark yellow	72	92-94	Dioxane
5		Pale-yellow	81	124-125	Acetone
6		Dark yellow	80	128-130	Dioxane
7		yellow	75	141-142	Ethanol

Table 2- Physical properties of polymers(8-12)

Polym. No.	Polymer structure	Conversion Ratio %	color	Softening Point C°
8		72	Dark yellow	148-160
9		69	Dark-brown	Gummy
10		66	yellow	Gummy
11		78	Dark-brown	90-110
12		71	Dark yellow	Gummy

It was noticeable that incorporation of bulk pendent group in the new polymers exhibit them good solubility in all organic solvents beside most of them are either gummy products or solids with low softening points and these properties are fitted with the expected ones besides these properties made the polymer suitable for many applications like coating, adhesives, molding and as plasticizers. Chemical structures of the prepared compounds are confirmed by FTIR spectral data and $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ spectra for some of them.

FTIR spectrum of compound (1) showed appearance of strong absorption bands at (1726 and 1184) cm^{-1} due to $\nu(\text{C=O})$ ester and $\nu(\text{C-O})$ ester respectively beside disappearance of $\nu(\text{O-H})$ carboxylic band all these points are excellent proofs for ester formation. Other bands shown in FTIR spectrum of compound (1) appeared at (1687,1629), 1595, 1309 cm^{-1} which are due to $\nu(\text{C=O})$ imide, $\nu(\text{C=C})$ and $\nu(\text{C-N})$ imide respectively [21].

FTIR spectrum of compound (2) showed appearance of clear absorption bands at (3380,3244 and 3120) cm^{-1} which are due to asym. $\nu(\text{NH}_2)$, sym. $\nu(\text{NH}_2)$ and $\nu(\text{NH})$ proving the success of hydrazide formation.

FTIR spectrum of compound (2) also showed absorption bands at 1629 cm^{-1} , 1605 cm^{-1} and 1240 cm^{-1} which are due to $\nu(\text{C=O})$ imide, $\nu(\text{C=O})$ amide and $\nu(\text{C-N})$ imide respectively [21].

FTIR spectra of Schiff bases (3-7) showed absorption bands at (1716-1760) cm^{-1} and (1640-1685) cm^{-1} due to asym. $\nu(\text{C=O})$ imide and sym. $\nu(\text{C=O})$ imide and bands at (1622-1666) cm^{-1} due to $\nu(\text{C=O})$ amide.

Also strong clear absorption bands appeared at (1600-1620) cm^{-1} due to $\nu(\text{C=N})$ imine while bands due to $\nu(\text{C=C})$, $\nu(\text{C-N})$ imide and $\nu(\text{NH})$ amide appeared at (1508-1587) cm^{-1} , (1301-1373) cm^{-1} and (3251-3494) cm^{-1} respectively. Other details of FTIR spectral data for Schiff bases (3-7) are listed in Table-3.

Table 3-FTIR spectral data (cm^{-1}) of Schiff bases (3-7)

Comp. No.	$\nu(\text{N-H})$	$\nu(\text{C-H})$ aromatic	$\nu(\text{C-H})$ aliphatic	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{C=C})$ aromatic	$\nu(\text{C-N})$ imide	Others
3	3423 3290	3030	2929 2860	1720 1676 1629	1600	1577	1330	$\nu(\text{NO}_2)$ 1508 1473
4	3494 3340	3033	2916 2858	1741 1685 1640	1600	1564	1357	p-sub. 819
5	3479	3005	2968 2839	1640 1622	1600	1508	1301	$\nu(\text{C-O})$ 1024 p-sub. 833
6	3452 3396	3055	2931 2850	1716 1658 1623	1610	1587	1328	—
7	3309 3251	3010	2933 2852	1760 1666	1620	1573	1373	$\nu(\text{O-H})$ 3380

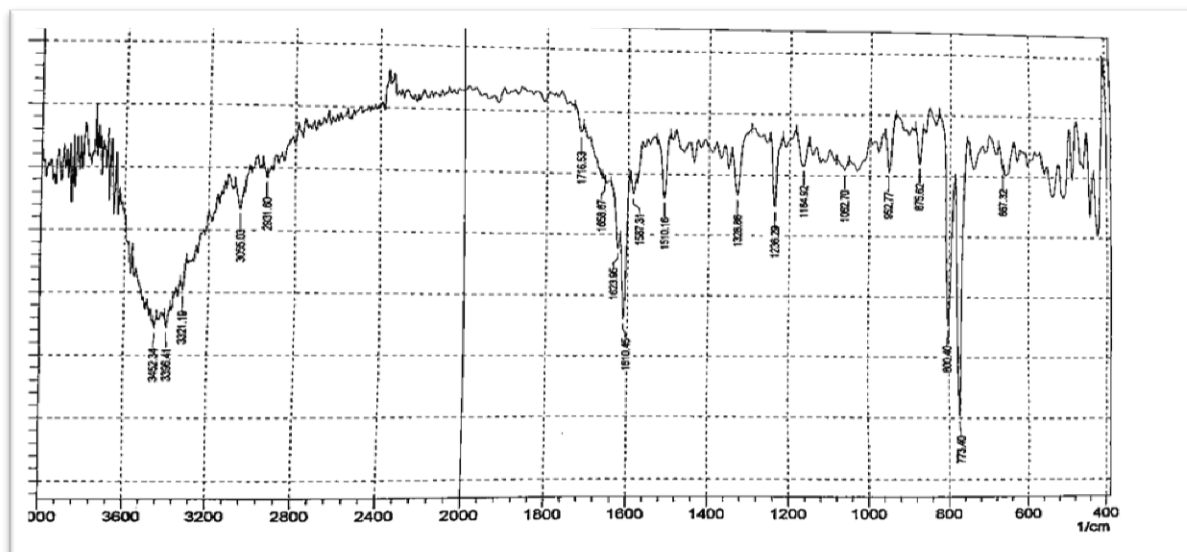


Figure 1- FTIR spectrum of compound (6).

$^1\text{H-NMR}$ spectrum of Schiff base (4) Figure-2 showed two singlet signals at ($\delta=2.26$ and 2.36)ppm belong to the protons of two methyl groups. Signal for vinylic protons appeared at ($\delta=6.5$)ppm and signals for aromatic protons and (NH)proton appeared at ($\delta=7.26-7.8$) and 7.82 ppm respectively [21].

$^{13}\text{C-NMR}$ spectrum of Schiff base(4) Figure-3 showed signals at ($\delta=14.84-15.07$)ppm and (21.37 ppm) belong to carbons of two methyl groups. Signals for vinylic and aromatic carbons appeared at($\delta=113.09-139.87$)ppm and signals for (C=N) and (C=O) carbons appeared at($\delta=153.90$ and 157.93) ppm.

$^1\text{H-NMR}$ spectrum of Schiff base (5) Figure-4 showed singlet signal at ($\delta=3.83$)ppm belong to OCH_3 protons. Signals for vinylic protons appeared at($\delta=7.05-7.07$)ppm and signals belong to aromatic protons and (NH) proton appeared at ($\delta=7.8-7.83$) ppm and signal at ($\delta=8.64$)ppm belong to (-CH=N-) proton.

$^{13}\text{C-NMR}$ spectrum of Schiff base(5) Figure-5 showed signal at($\delta=55.86$) belong to OCH_3 carbon and signals at ($\delta=114.56-132.29$) ppm belong to vinylic and aromatic carbons while signals belong to(C=N) and (C=O) carbons appeared at ($\delta=160.97$ and 162.14)ppm.

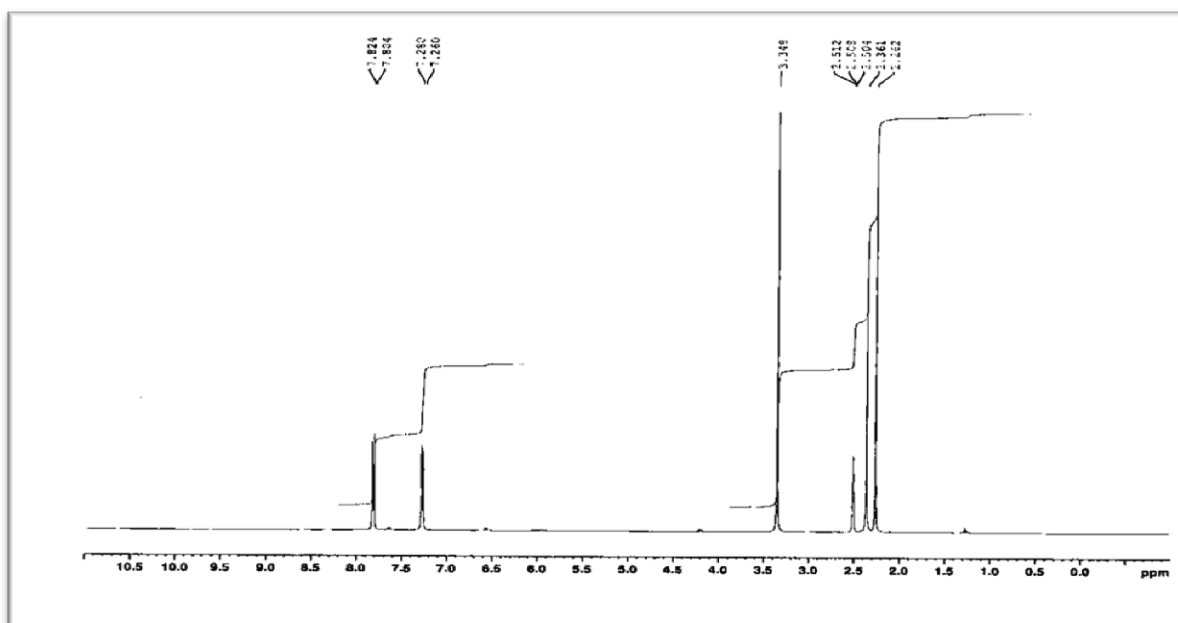


Figure 2- $^1\text{H-NMR}$ spectrum for compound (4).

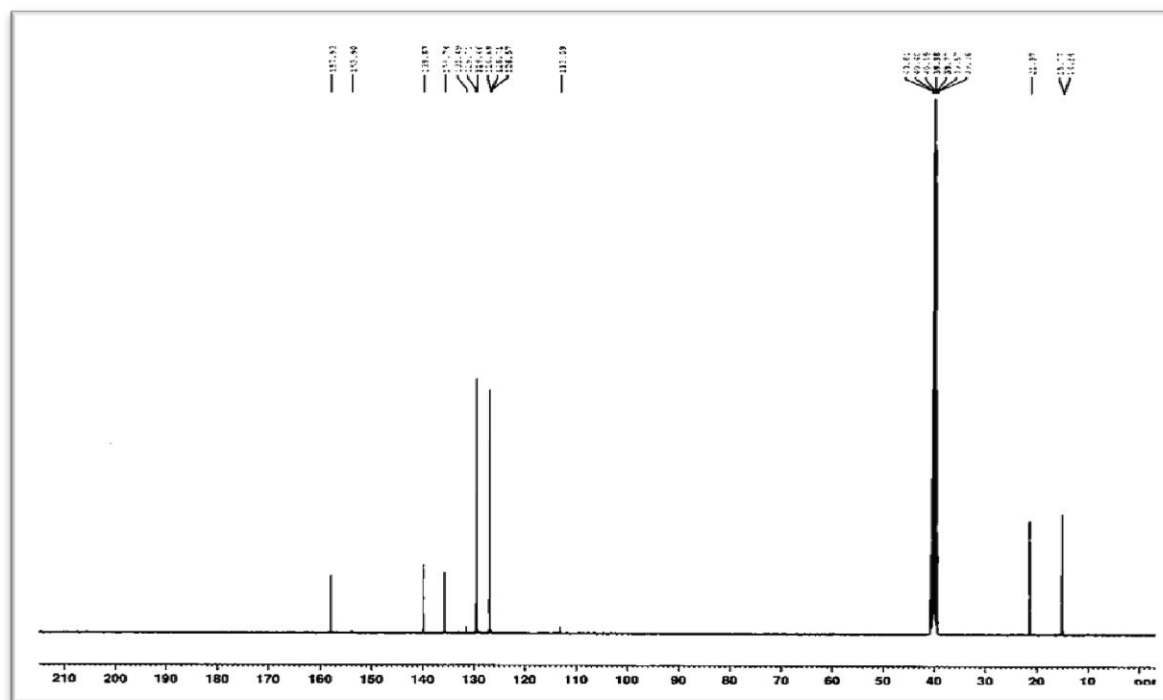


Figure 3- ^{13}C -NMR spectrum for compound (4).

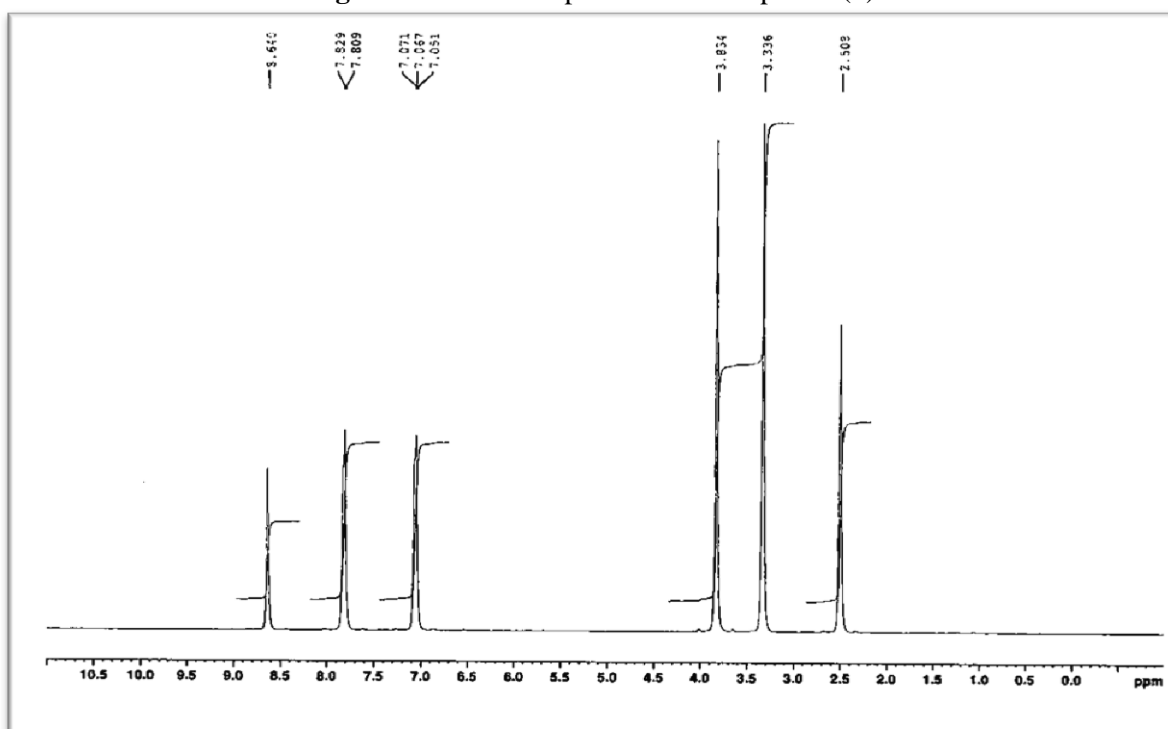


Figure 4- ^1H -NMR spectrum for compound (5) .

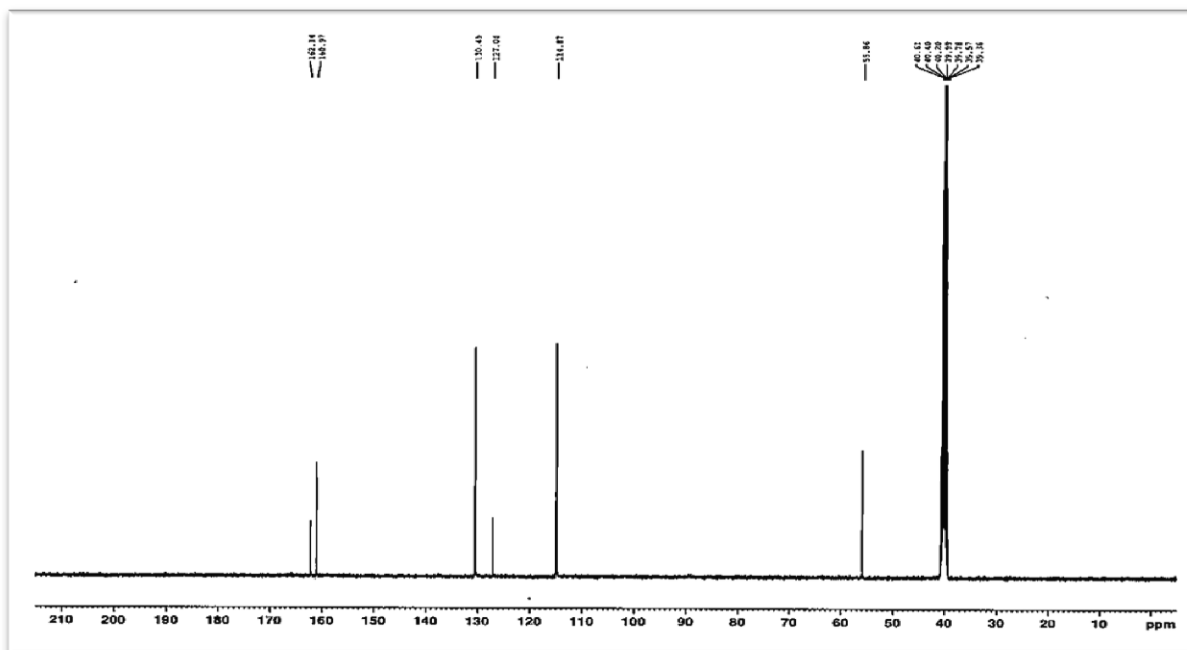


Figure 5- ^{13}C -NMR Spectrum for compound (5).

FTIR spectra of the prepared homopolymers(8-12) showed absorption bands at $(1654-1724)\text{cm}^{-1}$ and $(1625-1685)\text{cm}^{-1}$ due to $\nu(\text{C}=\text{O})$ imide and $\nu(\text{C}=\text{O})$ amide. A clear strong absorption bands appeared at $(1598-1622)\text{cm}^{-1}$ are due to $\nu(\text{C}=\text{N})$ imine, while absorption bands due to $\nu(\text{C}=\text{C})$ aromatic, $\nu(\text{C}-\text{N})$ imide and $\nu(\text{N}-\text{H})$ appeared at $(1510-1577)\text{cm}^{-1}$, $(1303-1386)\text{cm}^{-1}$ and $(3433-3244)\text{cm}^{-1}$ respectively. Details of FTIR spectral data of homopolymers (8-12) are listed in Table-4.

Table 4- FTIR spectral data (cm^{-1}) of homopolymers (8-12)

Polym. No.	$\nu(\text{N}-\text{H})$	$\nu(\text{C}-\text{H})$ aromatic	$\nu(\text{C}-\text{H})$ aliphatic	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}=\text{C})$ aromatic	$\nu(\text{C}-\text{N})$ imide	Others
8	3377 3286	3040	2925 2856	1720 1633	1598	1577	1328	$\nu(\text{NO}_2)$ 1514 1470
9	3420 3380	3040	2920 2854	1724 1680 1658	1604	1514	1363	p-sub. 819
10	3361 3244	3070	2931 2840	1654 1625	1602	1510	1303	$\nu(\text{C}-\text{O})$ 1026 p-sub. 833
11	3433 3361	3055	2940 2875	1710 1685	1608	1510	1325	—
12	3420 3380	3041	2921 2850	1680	1622	1571	1386	$\nu(\text{O}-\text{H})$ 3420

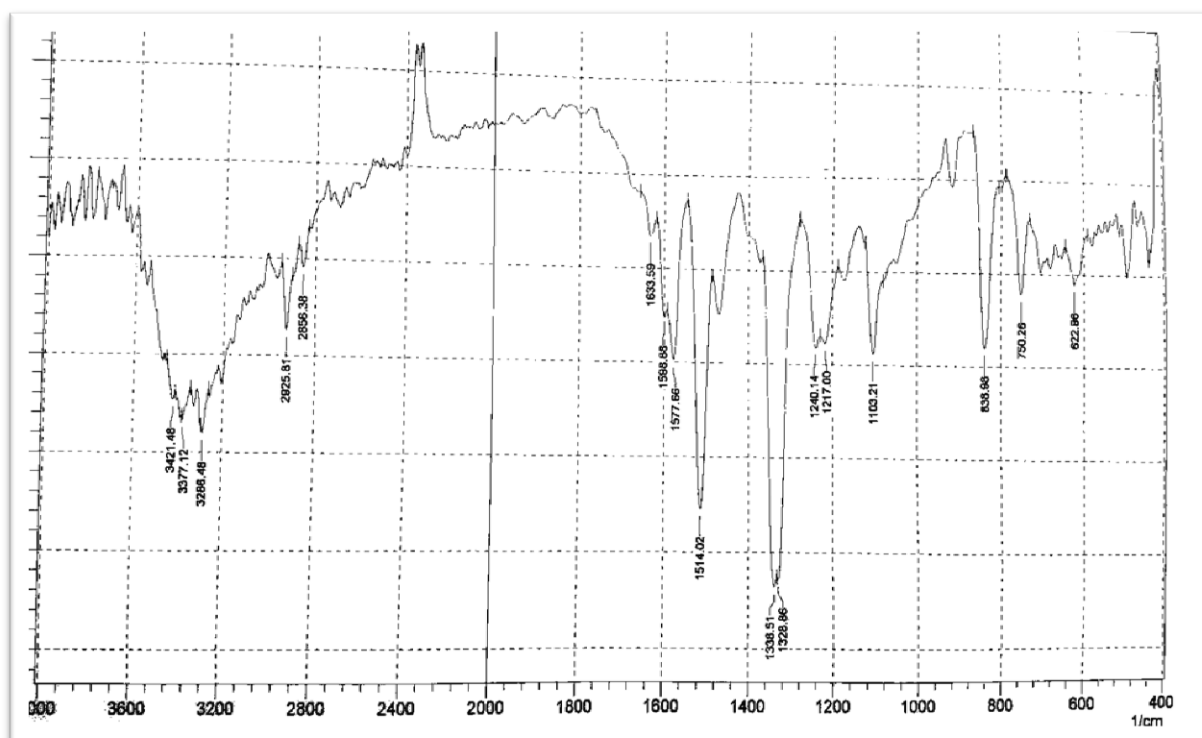


Figure 6- FTIR spectrum of compound (8).

$^1\text{H-NMR}$ spectrum of homopolymer (10) showed signals at ($\delta=1.56$)ppm and (3.89)ppm belong to aliphatic protons in imide ring and OCH_3 protons respectively. Signals belong to aromatic protons appeared at ($\delta=6.95$ -7.28) ppm while signals belong to (NH) proton and ($-\text{HC}=\text{N}-$) proton appeared at ($\delta=7.8$ and 8.63)ppm.

$^{13}\text{C-NMR}$ spectrum of homopolymer (10) showed signals at ($\delta=29.71$)ppm belong to aliphatic carbons in imide ring, signal at ($\delta=55.48$)ppm belong to OCH_3 carbon, while signals belong to aromatic carbons appeared at ($\delta=114.17$ -130.29)ppm and signals belong to ($\text{C}=\text{N}$) and ($\text{C}=\text{O}$) carbons appeared at ($\delta=161.01$ and 166.65)ppm respectively [21].

The second strategy used in this work for producing polymaleimides with improved properties is copolymerization since copolymerization is an efficient method for introducing new units to polymaleimide back bone chains leading to exhibit the resulted copolymers new properties [14].

Thus the prepared maleimide monomers (3-7) were introduced in free radical chain growth copolymerization with acrylonitrile producing new copolymaleimides (13-17) with new properties [15]. The incorporation of acrylonitrile units in polymeric chains of the new copolymers (13-17) exhibit them good solubility in most organic solvents but it was noticeable that their softening points in general are higher than those belong to the corresponding homopolymers and this is due to the presence of polar cyano groups ($\text{C}\equiv\text{N}$) in acrylonitrile units which increased inter chain forces leading to increase softening points. Physical properties of copolymers(13-17) are shown in Table-5 .

FTIR spectra of the new copolymers(13-17) showed very important clear and strong absorption band at (2243cm^{-1}) due to $\nu(\text{C}\equiv\text{N})$ and this is an excellent proof for success of copolymerization reaction [21].

Other absorption bands appeared at (1685 - 1724) cm^{-1} , (1620 - 1683) cm^{-1} , (1596 - 1620) cm^{-1} , (1512 - 1573) cm^{-1} and (1303 - 1371) cm^{-1} which are attributed to $\nu(\text{C}=\text{O})$ imide, $\nu(\text{C}=\text{O})$ amide, $\nu(\text{C}=\text{N})$ imine, $\nu(\text{C}=\text{C})$ aromatic and $\nu(\text{C}-\text{N})$ imide respectively.

Details of FTIR spectral data for copolymers (13-17) are listed in Table-6.

Table 5- Physical properties of copolymers(13-17)

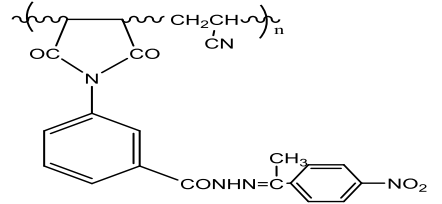
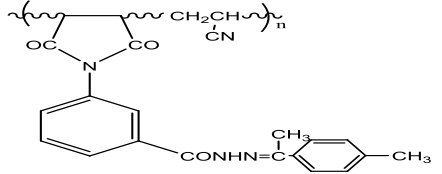
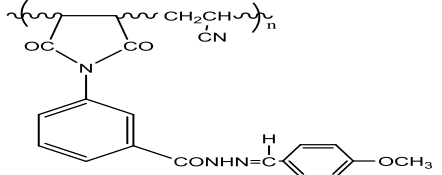
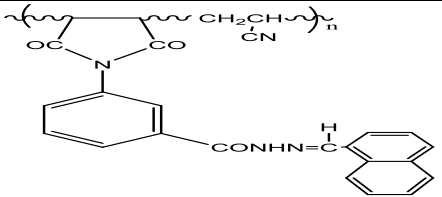
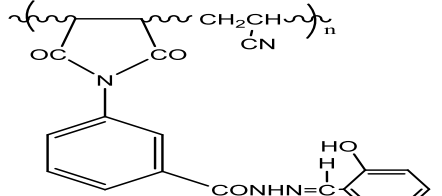
Polym. No.	Polymer structure	Conversion Ratio %	Color	Softening point
13		70	brown	160-172
14		74	brown	105-115
15		65	yellow	130-145
16		76	Dark yellow	110-120
17		68	brown	120-135

Table 6- FTIR spectral data (cm⁻¹) of copolymers (13-17)

Polym. No.	$\nu(\text{N-H})$	$\nu(\text{C-H})$ aromatic	$\nu(\text{C-H})$ aliphatic	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{C=C})$ aromatic	$\nu(\text{C-N})$ imide	$\nu(\text{C=N})$	Others
13	3236	3080	2929 2860	1690 1656	1596	1521	1346	2243	$\nu(\text{NO}_2)$ 1521 1454
14	3390	3010	2941 2860	1685 1630	1602	1562	1361	2243	p-sub. 823
15	3411	3080	2929 2871	1712 1660 1620	1602	1512	1303	2243	$\nu(\text{C-O})$ 1026 p-sub.
16	3446 3386	3049	2958 2885	1724 1683 1654	1608	1541	1326	2243	—
17	3400 3320	3020	2941 2877	1710 1680	1620	1573	1371	2243	$\nu(\text{O-H})$ 3400

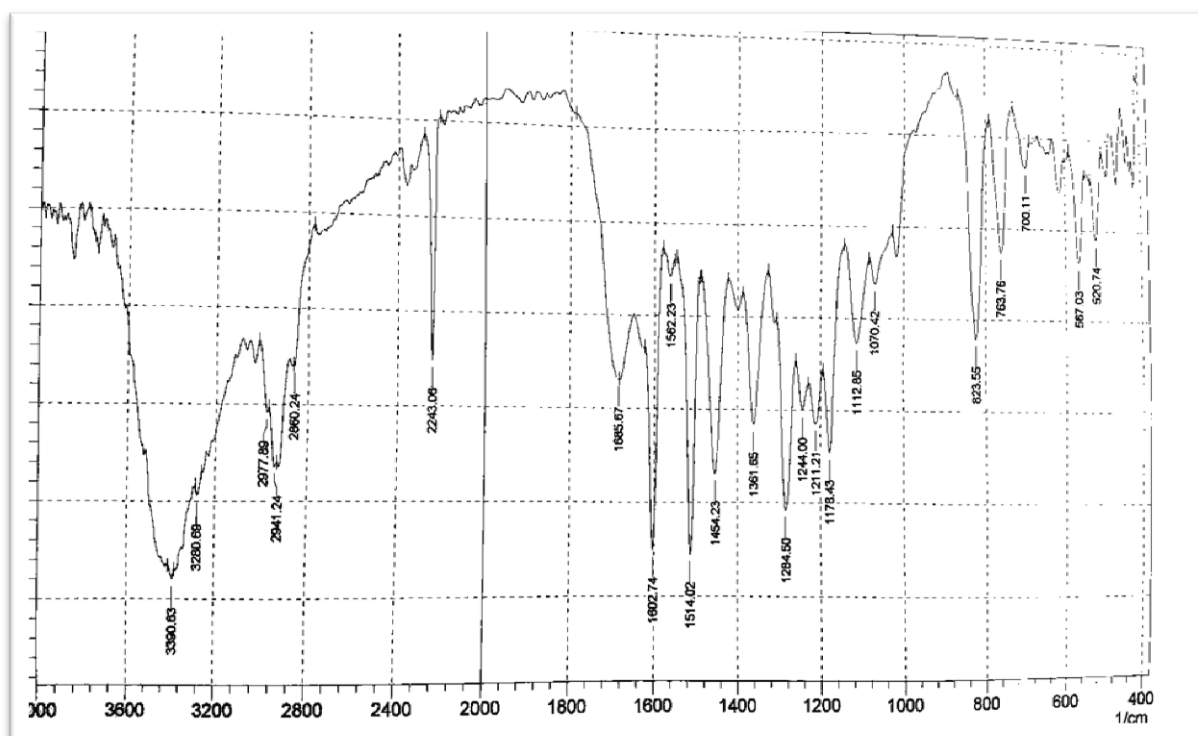


Figure 7- FTIR spectrum of compound (14).

$^1\text{H-NMR}$ spectrum of copolymer (13) showed signals at ($\delta=0.9-1.3$)ppm belong to ($-\text{CH}_2-\text{CHCN}-$) protons, signal at ($\delta=2.2$)ppm belong to CH_3 protons and signal at ($\delta=1.65$)ppm belong to aliphatic protons in imide ring. Signals for aromatic protons appeared at ($\delta=7.0-8.36$)ppm and signal belong to (NH) proton appeared at ($\delta=8.73$)ppm.

$^{13}\text{C-NMR}$ spectrum of copolymer(13) showed signals at ($\delta=14.75-15.59$) ppm,(22.21),(25.6-27.88)ppm and (28.34-33.25)ppm which are belong to (CH_2 -) carbon, CH_3 carbon, ($\text{C}\text{H}\text{CN}$) and aliphatic carbons in imide ring respectively. Other signals appeared at ($\delta=68.74$) and ($\delta=120.14-131.18$) ppm belong to ($\text{C}\equiv\text{N}$) carbon and aromatic carbons while signals belong to ($\text{C}=\text{N}$) and ($\text{C}=\text{O}$) carbons appeared at ($\delta=150.0-152.5$ and 160)ppm.

$^1\text{H-NMR}$ spectrum of copolymer (15) Figure-8 showed signals at ($\delta=1.4-1.7$) and 2.14ppm belong to (CH_2) protons,(CHCN) and protons in imide ring respectively. Signals belong to (OCH_3) protons and aromatic protons appeared at ($\delta=3.83$) and (6.55-7.8)ppm while signals belong to (NH) proton and ($-\text{CH}=\text{N}-$) proton appeared at ($\delta=8.61$ and 8.64)ppm.

$^{13}\text{C-NMR}$ spectrum of copolymer (15) Figure-9 showed signals at ($\delta=14.76-27.2$) ppm belong to (CH_2) and ($-\text{CHCN}$) carbons and signals at($\delta=28.3-33.24$)ppm belong to aliphatic carbons in imide ring. Signals belong to OCH_3 carbon and ($\text{C}\equiv\text{N}$) carbon appeared at ($\delta=55.75-55.86$)ppm and (67.49-68.44)ppm , signals for aromatic carbons appeared at ($\delta=114.87-130.45$)ppm and signals belong to($\text{C}=\text{N}$) and ($\text{C}=\text{O}$) carbons appeared at ($\delta=150.19$, 160.97 and 162.14)ppm.

Finally $^1\text{H-NMR}$ spectrum of copolymer (17) showed signals at ($\delta=1.44,1.75$)ppm and (2.1) ppm which belong to (CH_2),(CHCN) and aliphatic protons in imide ring respectively. Signals for aromatic protons appeared at ($\delta= 6.98-7.4$)ppm ,signals for (NH) proton at ($\delta=7.7$)ppm and signal for ($-\text{CH}=\text{N}-$) proton at ($\delta=9.0$)ppm.

$^{13}\text{C-NMR}$ spectrum of copolymer (17) showed signals at ($\delta=14.76-22.51$) ppm belong to ($-\text{C}\text{H}_2-$), (25.51-27.88)ppm belong to ($-\text{C}\text{H}\text{CN}-$) and at ($\delta= 28.36-33.26$)ppm belong to carbon atoms in imide ring.

Other signals appeared at ($\delta=67.49$)ppm and (117-133.72)ppm belong to($\text{C}\equiv\text{N}$) carbon and aromatic carbons while signals belong to ($\text{C}=\text{N}$) and ($\text{C}=\text{O}$) carbons appeared at ($\delta=159.11-163.26$)ppm.

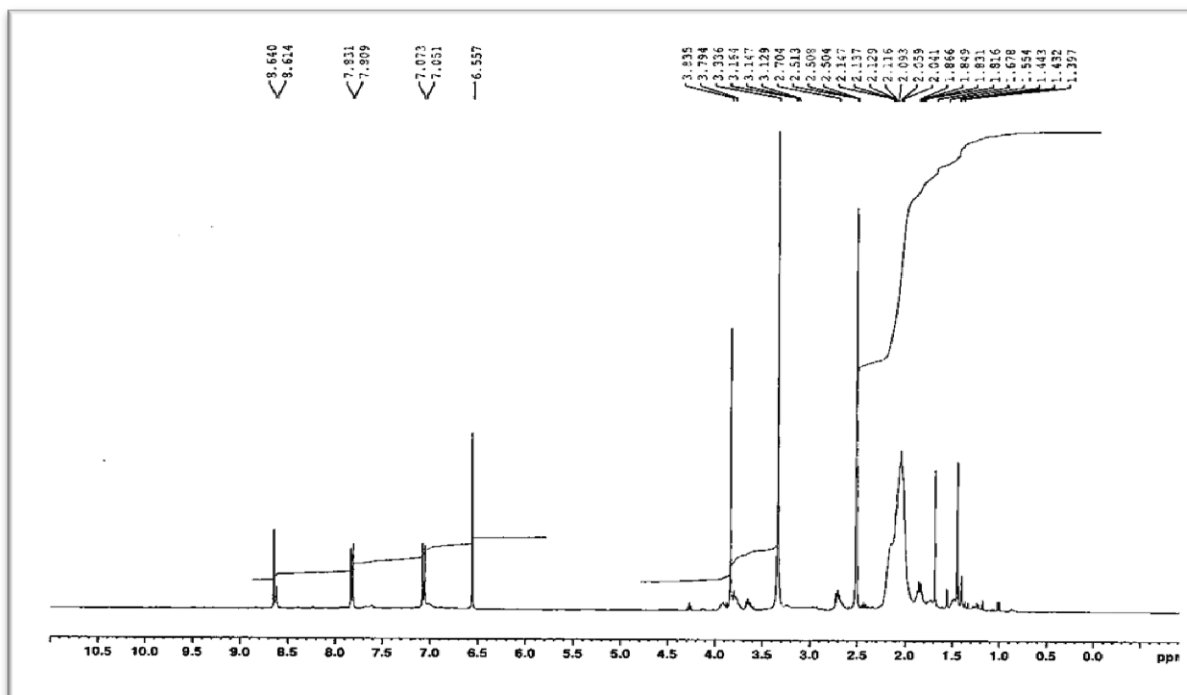


Figure 8- $^1\text{H-NMR}$ spectrum for compound (15).

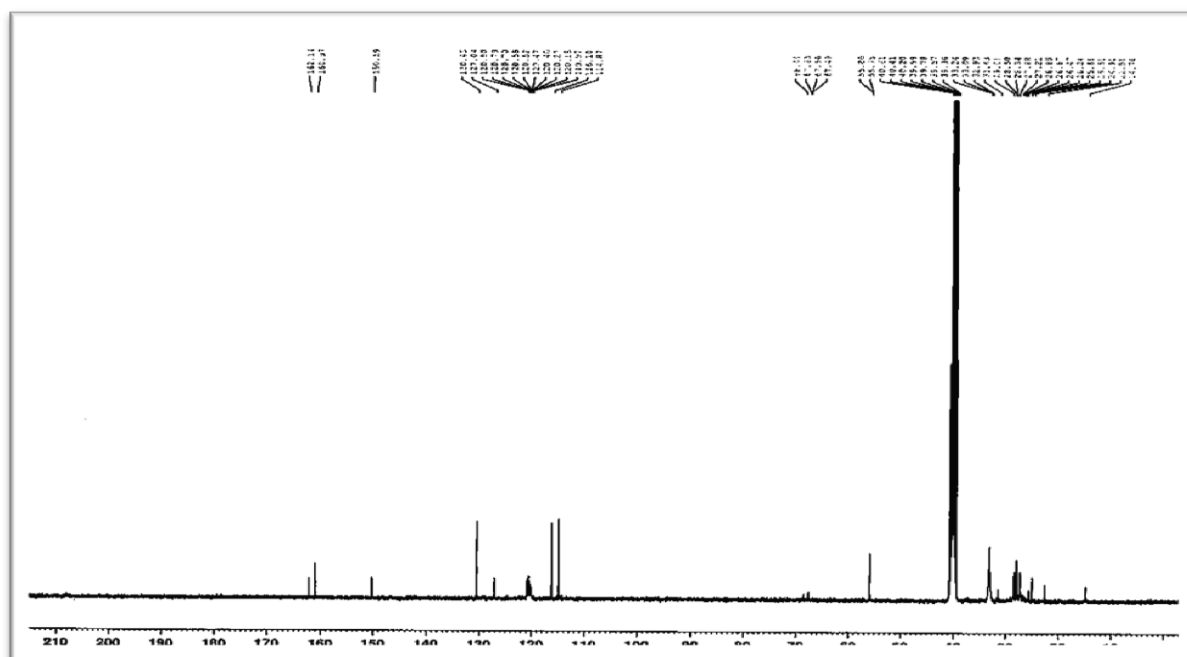


Figure 9- $^{13}\text{C-NMR}$ spectrum for compound (15).

Conclusion

Modification of polymaleimides properties were performed first through introducing of bulk pendent Schiff base moieties in repeating units of polymaleimide and second by copolymerization. The two used methods improved physical properties of the prepared polymers.

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