



Synthesis and Characterization of Several New Copolymers Based on Maleimides Bearing 1, 3, 4-Oxadiazole Moiety

Ahlam Marouf Al-Azzawi¹, Hiba Khadim Yaseen^{2*}

¹Department of Chemistry, College of Science, University of Baghdad, Baghdad, Iraq

²Ministry of Science and Technology, Baghdad, Iraq

Abstract

A series of new copolyimides containing pendant 1,3,4-oxadiazole moiety were synthesized via multisteps. In the first step five N-(5-substituted-1,3,4-oxadiazole-2-yl)maleamic acids were prepared via reaction of maleic anhydride with 2-amino-5-substituted-1,3,4-oxadiazoles. The obtained amic acids were dehydrated in the second step affording the corresponding N-(5-substituted-1,3,4-oxadiazole-2-yl) maleimides. In the third step the newly synthesized maleimides were introduced successfully in free radical copolymerization reaction with four vinylic monomers including acrylo nitrile, methacrylonitrile, methyl acrylate and methyl meth acrylate respectively producing twelve new copolymers having different physical properties which may serve different applications. The new copolymers are of great importance since it contain two important components (maleimide and 1,3,4-oxadiazole) cycles in their repeating units which exhibit good thermal and chemical stability beside the presence of acrylo nitriles or acrylates components which exhibit better solubility and processing properties.

Keywords: Maleamic acids, Maleimides, Copolymerization, 1,3,4-oxadiazole

تحضير وتشخيص عدد من البوليمرات المشتركة الجديدة اعتماداً على مالي ايميدات حاملة لمكونة 1،3،4-اوksادايازول

احلام معروف العزاوي¹، هبة كاظم ياسين^{2*}

¹ قسم الكيمياء ، كلية العلوم، جامعة بغداد ، بغداد - العراق

² وزارة العلوم والتكنولوجيا ، بغداد - العراق

الخلاصة

تضمن البحث تحضير عدد من البوليمرات المشتركة الجديدة للايميدات الحلقية تحتوي على مجاميع متدلالية من 1،3،4-اوksادايازول وذلك من خلال انجاز عدة خطوات. تضمنت الخطوة الاولى تحضير خمسة من N-(5-معوض-1،3،4-اوksادايازول-2-يل) حوامض المالي اميك: وذلك من تفاعل انهيدريد الماليك مع مركبات 2-امينو-5-معوض-1،3،4-اوksادايازول. في الخطوة الثانية تم سحب الماء من حوامض المالي اميك المحضرة وبذلك تم الحصول على N-(5-معوض-1،3،4-اوksادايازول-2-يل) مالي ايميدات المقابلة. اما في الخطوة الثالثة فقد تم ادخال المالي ايميدات المحضرة في تفاعل بلمرة مشتركة

* Email: hiba_k85@yahoo.com

منجزة بالجذور الحرة مع عدد من المونوميرات الفايينيلية والتي تضمنت اكريلونايترايل، ميث اكريلونايترايل،
 مثيل اكريلات ومثيل ميث اكريلات على التوالي مما اسفر عن تحضير اثنا عشر بوليمر مشترك جديد
 بمواصفات فيزيائية مختلفة مما يخدم تطبيقات مختلفة.
 تعتبر البوليمرات المشتركة المحضرة في هذا البحث غاية في الاهمية نظراً لاحتواء وحداتها المتكررة على
 مكونتين مهمتين هما حلقة المالبي ايميد وحلقة 1,3,4-اوksاديازول والتي كل منهما تمنح البوليمر زيادة في
 الاستقرار الحراري والكيميائي فضلا عن وجود مكونة الاكريلونايترايل او الاكريلات في الوحدات المتكررة والتي
 تمنح قابلية ذوبان افضل وصفات تصنيعية افضل للبوليمرات المشتركة المحضرة.

Introduction

Polyimides are an important class of materials featuring high thermal stability, chemical resistance and excellent mechanical properties [1-3]. Besides they maintain an excellent balance of electrical, thermal, mechanical, physical and chemical properties over a wide range of temperatures [4, 5]. However they are usually difficult in direct processing due to their high glass transition and melting temperatures and their limited solubility which limit their usefulness for many applications. For this reason significant efforts have been made to improve their properties [6-8] by chemical modification of the chain backbone through copolymerization [9-13]. Thus in the present work a series of new copolymers were synthesized by preparation of new maleimide monomers bearing 1,3,4-oxadiazole moiety then introducing these monomers in free radical copolymerization with selected vinylic monomers producing new polyimides having new improved properties which may serve different applications.

Experimental

All chemicals employed were purchased from BDH, Merck and Flucka chemical companies and were used without further purification. Uncorrected melting points were determined on Thomas Hoover apparatus. FTIR spectra were recorded on SHIMADZU FTIR-8400 Fourier Transform Infrared spectrophotometer as KBr disc. ¹H-NMR and ¹³C-NMR spectra were recorded on Bruker 300 MHz instrument using DMSO-d₆ as solvent and TMS as internal reference. Softening points were determined on thermal microscope Riecher thermover.

1- Preparation of N-(5-substituted-1, 3, 4-oxadiazole-2-yl) maleamic acids (1-5)

The titled amic acids were prepared via reaction of equimolar amounts of maleic anhydride and 2-amino-5-substituted-1,3,4-oxadiazoles according to literature procedures [14].

Purification was performed by recrystallization from ethanol or acetone. Physical properties and FTIR spectral data of amic acids (1-5) are shown in Table-1.

2- Preparation of N-(5-substituted-1,3,4-oxadiazole-2-yl) maleimides (6-10)

The titled maleimides were prepared by dehydration of maleamic acids (1-5) using fusion technique according to literatures [15].

The obtained maleimides were purified by recrystallization from dioxane, cyclohexane, acetone and their physical properties and FTIR spectral data are shown in Table- 2.

3- Preparation of [N-(5-substituted-1, 3,4-oxadiazole-2-yl) maleimide-acrylo nitrile] Copolymers (11-14)

In a screw capped polymerization bottle equimolar amounts (0.001 mol) of N-(5-substituted-1,3,4-oxadiazole-2-yl)maleimide and acrylonitrile were dissolved in (12 mL) of THF then (0.0002g) of AIBN (azobisisobutyronitrile) was added (as initiator) [9]. The bottle was flushed with nitrogen for few minutes then firmly stoppered and maintained at (75°C) for 3 hrs. The resulted solution was poured into (25 mL) of methanol and the precipitated polymer was filtered, washed with methanol and dried. Physical properties of copolymers (11-14) are shown in Table- 3.

4- Preparation of [N-(5-substituted-1, 3,4-oxadiazole-2-yl) maleimide-methacrylo nitrile] Copolymers (15,16)

The titled copolymers were synthesized by following the same procedure steps used in preparation of copolymers (11-14) except using of methacrylo nitrile instead of acrylo nitrile. Physical properties of copolymers (15, 16) are shown in Table- 3.

5- Preparation of [N-(5-subsituted-1,3,4-oxadiazole-2-yl)maleimide-methyl acrylate] copolymers (17,18)

The titled copolymers (17, 18) were prepared by following the same procedure steps used in preparation of copolymers (11-14) except using of methyl acrylate instead of acrylo nitrile. Physical properties of copolymers (17, 18) are shown in Table- 4.

6- Preparation of [N-(5-subsituted-1,3,4-oxadiazole-2-yl) maleimide-methyl meth acrylate] Copolymers (19-22)

The titled copolymers (19-22) were prepared by following the same procedure steps used in preparation of copolymers (11-14) except using of methyl meth acrylate instead of acrylo nitrile. Physical properties of copolymers (19-22) are shown in Table- 4.

Table 1- Physical properties and FTIR spectral data of amic acids (1-5)

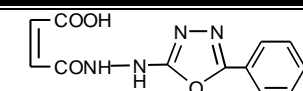
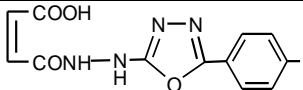
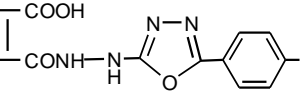
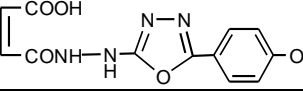
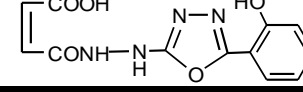
Comp. No.	Compound structure	Color	Yield %	Melting point °C	FTIR Absorption bands (cm ⁻¹)						
					$\nu(\text{O-H})$ (N-H)	$\nu(\text{C-H})$ aromatic	$\nu(\text{C=O})$ carbox.	$\nu(\text{C=O})$ amide	$\nu(\text{C=N})$	$\nu(\text{C=C})$ aromatic	$\nu(\text{CO})$
1		White	83	108-110	3298 3122	3062	1716	1652	1618	1596	1199 1024
2		Off white	70	130-131	3420 3240	3063	1724	1674	1620	1600	1230 1026
3		White	80	134-136	3276 3110	3101	1714	1658	1616	1581	1203 1101 1047
4		Off white	78	140-141	3434 3367	3058	1706	1691	1639	1593	1272 1174
5		Faint yellow	76	124-126	3440 3332	3076	1726	1704	1635	1610	1207 1178 1029

Table 2- Physical properties and FTIR spectral data of maleimides (6-10)

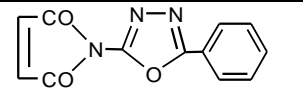
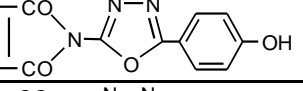
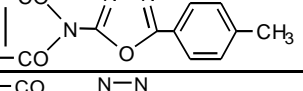
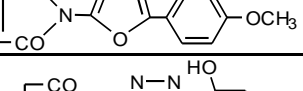
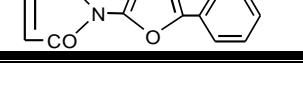
Comp. No.	Compound structure	Color	Yield %	Melting point °C	FTIR Absorption bands (cm ⁻¹)					
					$\nu(\text{C-H})$ aromatic	$\nu(\text{C=O})$ imide	$\nu(\text{C=N})$	$\nu(\text{C=C})$ aromatic	$\nu(\text{C-N})$ imide	$\nu(\text{C-O})$
6		Off white	85	163-165	3035	1789 1733	1650	1600	1336	1176 1024
7		Deep yellow	81	178-180	3058	1706	1637	1585	1348	1265 1151
8		Yellow	88	166-168	3026	1780 1733	1612	1573	1290	1186 1141
9		Off white	72	148-150	3058	1722	1656	1606	1357	1261 1178 1022
10		Yellow	77	140-142	3058	1706	1637	1589	1348	1265 1164

Table 3- Physical properties of copolymers (11-16)

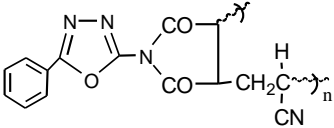
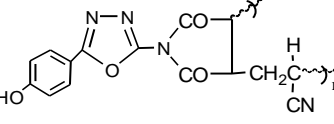
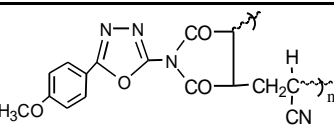
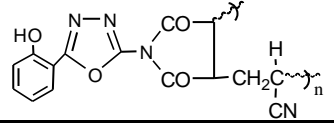
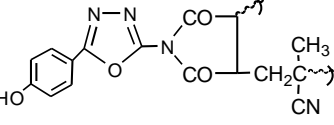
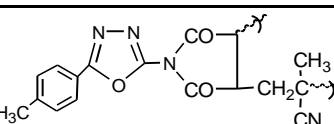
Comp. No.	Compound structure	Color	Conv. %	Soft. point °C	Solubility							
					THF	DMSO	DMF	MeOH	Dioxane	Acetone	CHCl ₃	C ₆ H ₆
11		Off white	80	195-207	Sh	S	S	ins	Ins	Sh	Sh	ins
12		Gray	66	202-212	Ps	S	S	ins	Ps	Sh	Sh	ins
13		Faint yellow	74	198-205	Sh	S	S	ins	Ins	Sh	Sh	ins
14		Faint gray	65	215-225	Ps	S	S	ins	Ps	Sh	Sh	ins
15		Yellow	71	190-200	Sh	S	S	ins	Ins	Sh	Sh	ins
16		Gray	68	205-210	Sh	S	S	ins	Ins	Sh	Sh	ins

Table 4- Physical properties of copolymers (17-22)

Comp. No.	Compound structure	Color	Conv. %	Soft. point °C	Solubility							
					THF	DMF	DMSO	MeOH	Dioxane	Acetone	CHCl ₃	C ₆ H ₆
17		Faint yellow	63	Gummy	S	S	S	ins	S	S	S	ins
18		Gray	69	Gummy	S	S	S	ins	S	S	S	ins
19		Gray	72	165-177	S	S	S	ins	Ps	S	S	ins
20		Gray	78	145-153	S	S	S	ins	Ps	S	S	ins
21		Off white	77	Gummy	S	S	S	ins	S	S	S	ins
22		Faint yellow	67	Gummy	S	S	S	ins	S	S	S	ins

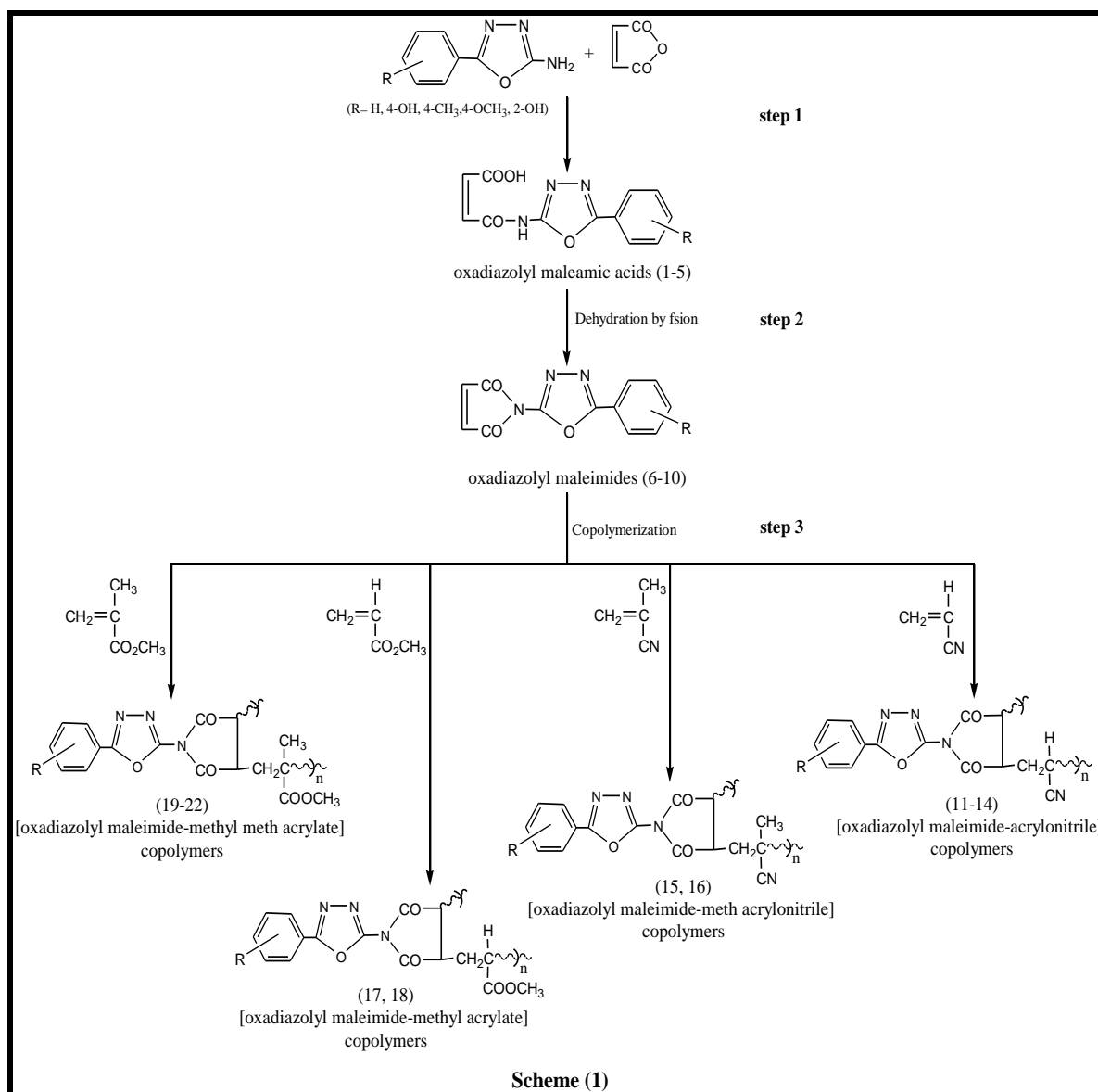
S=soluble, ins=insoluble, Ps=partial soluble, Sh=soluble in hot solvent

Results and Discussion

In general improvement of polymers physical and mechanical properties can be performed via modification of polymers either by using additives or by copolymerization. Thus significant efforts have been made to improve polymer physical properties by copolymerization which combined between different monomers together leading to chemical modification of the back bone chain producing new polymers having new physico-chemical properties depending on the nature of the introduced commoners [9].

In light of these observations we planned in this work to synthesize a variety of new copolymers based on new maleimide monomers bearing 1,3,4-oxadiazole moiety.

Performing this target was made by many steps which are summarized in Scheme-1.



In the first step a series of five 2-amino-5-substituted-1,3,4-oxadiazoles were prepared according to literature procedures [16] via reaction of equimolar amounts of semicabazide hydrochloride with different aromatic aldehydes producing semicarbazones which inturn introduced in oxidative cyclization under the influence of bromine and anhydrous sodium acetate in glacial acetic acid producing the target oxadiazoles.

Physical properties and spectral data of the prepared oxadiazoles are fitted with those reported in literatures. In the second step five new maleamic acids were prepared via reaction of the prepared oxadiazoles with maleic anhydride. Physical properties and FTIR spectral data of the prepared maleamic acids are listed in Table-1. Dehydration of the prepared maleamic acids (1-5) by fusion technique afforded the corresponding maleimides (6-10) which their physical properties and FTIR spectral data are listed in Table-2. FTIR spectra of maleamic acids showed clear absorption bands at $(3276-3440) \text{ cm}^{-1}$ and $(3110-3367) \text{ cm}^{-1}$ due to $\nu(\text{O-H})$ carboxyl and $\nu(\text{N-H})$ amide respectively while these bands were disappeared in FTIR spectra of the corresponding maleimides beside the appearance of asym. and sym. $\nu(\text{C=O})$ imide absorption bands at $(1780-1789) \text{ cm}^{-1}$ and $(1706-1733) \text{ cm}^{-1}$ indicated the success of dehydration reaction [17].

Introducing of the newly synthesized oxadiazolyl maleimides (6-10) in chain growth free radical copolymerization reaction with acrylonitrile and meth acrylonitrile afforded the new copolymers (11-14) and (15, 16) respectively while free radical copolymerization of maleimides (6-10) with methyl

acrylate and methyl meth acrylate afforded the new copolymers (17, 18) and (19-22) respectively. The prepared copolymers showed different physical properties which are shown in Tables-3 and -4.

FTIR spectra of copolymers (11-16) showed a clear absorption band at (2235-2244) cm^{-1} due to $\nu(\text{C}\equiv\text{N})$ and this proved the presence of acrylonitrile or meth acrylonitrile units these copolymers [17]. Other absorption bands appeared at (1701-1731) cm^{-1} , (1639-1685) cm^{-1} , (1583-1612) cm^{-1} and (1305-1398) cm^{-1} which are attributed to $\nu(\text{C}=\text{O})$ imide, $\nu(\text{C}=\text{N})$ oxadiazole, $\nu(\text{C}=\text{C})$ aromatic and $\nu(\text{C}-\text{N})$ imide respectively.

$^1\text{H-NMR}$ spectrum of copolymer (12) showed signal at ($\delta=1.2$) ppm belong to (CH_2) protons and signals at ($\delta=2.7$) ppm belong to aliphatic protons in imide ring and ($-\text{CHCN}$) proton. Signals belong to aromatic protons appeared at ($\delta=6.7-7.8$) ppm and signal for OH proton appeared at ($\delta=9.7$) ppm. $^{13}\text{C-NMR}$ spectrum of copolymer (12) showed signal at ($\delta=23.35$) ppm belong to CH_2 carbon and signal at ($\delta=24.42$) ppm belong to aliphatic carbons in imide ring and ($-\text{CHCN}$) carbon.

Signals for aromatic carbons appeared at ($\delta=123.42-131.57$) ppm while signals for ($\text{C}\equiv\text{N}$), ($\text{C}=\text{N}$) and ($\text{C}=\text{O}$) imide carbons appeared at ($\delta=157.41, 160.49$ and 168.04) ppm respectively [18].

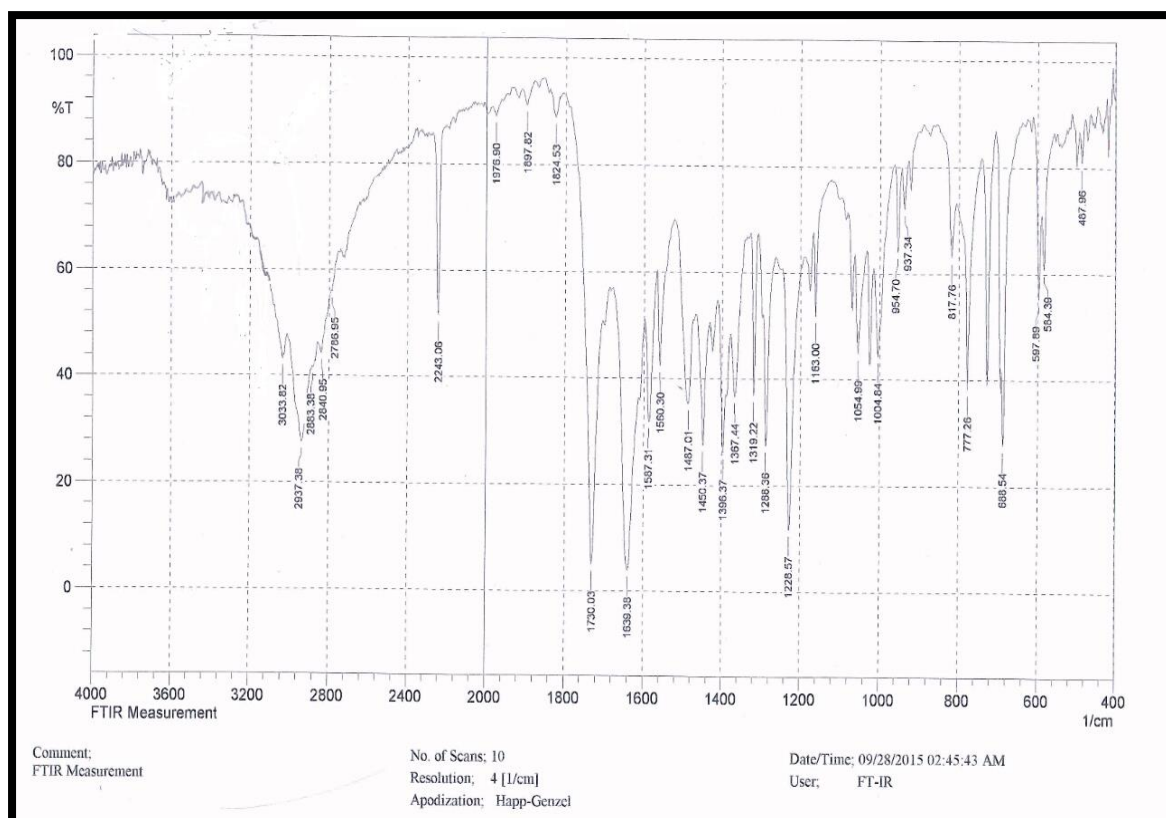
On the other hand FTIR spectra of copolymers (17-22) revealed clear absorption bands at (1728-1735) cm^{-1} due to $\nu(\text{C}=\text{O})$ ester and other absorption bands at (1703-1724) cm^{-1} , (1639-1687) cm^{-1} , (1587-1612) cm^{-1} and (1367-1398) cm^{-1} which are attributed to $\nu(\text{C}=\text{O})$ imide, $\nu(\text{C}=\text{N})$ oxadiazole, $\nu(\text{C}=\text{C})$ aromatic and $\nu(\text{C}-\text{N})$ imide respectively. Other details for FTIR spectral data of copolymers (11-16) and (17-22) are listed in Tables -5 and -6.

Table 5- FTIR spectral data (cm^{-1}) of copolymers (11-16)

Comp. No.	$\nu(\text{C}-\text{H})$ aromatic	$\nu(\text{C}-\text{H})$ aliphatic	$\nu(\text{C}\equiv\text{N})$	$\nu(\text{C}=\text{O})$ imide	$\nu(\text{C}=\text{N})$ oxadiazole	$\nu(\text{C}=\text{C})$ aromatic	$\nu(\text{C}-\text{N})$ imide	$\nu(\text{C}-\text{O})$	Others
11	3033	2937 2840	2243	1730	1639	1587	1319	1228 1163 1054	-
12	3068	2985 2925	2243	1701	1650	1583	1365	1238 1159 1012	$\nu(\text{O}-\text{H})$ 3496, 3382 $\nu(\text{p-sub.})$ 823
13	3080	2937 2840	2243	1731	1654	1608	1305	1259 1178 1024	$\nu(\text{p-sub.})$ 844
14	3118	2931	2244	1712	1645	1585	1360	1224 1170 1016	$\nu(\text{O}-\text{H})$ 3440, 3396
15	3064	2985 2920	2235	1712	1683	1591	1398	1236 1141 1010	$\nu(\text{O}-\text{H})$ 3490, 3386 $\nu(\text{p-sub.})$ 821
16	3045	2974 2860	2239	1720	1685	1612	1369	1284 1182 1093	$\nu(\text{p-sub.})$ 831

Table 6- FTIR spectral data (cm^{-1}) of copolymers (17-22)

Comp. No.	$\nu(\text{C-H})$ aromatic	$\nu(\text{C-H})$ aliphatic	$\nu(\text{C=O})$ ester	$\nu(\text{C=O})$ imide	$\nu(\text{C=N})$ oxadiazole	$\nu(\text{C=C})$ aromatic	$\nu(\text{C-N})$ imide	$\nu(\text{C-O})$	Others
17	3050	2987 2939	1731	1716	1647	1600	1371	1272 1178 1022	$\nu(\text{p-sub.})$ 820
18	3002	2952 2887	1728	1705	1639	1587	1398	1242 1193 1151	$\nu(\text{O-H})$ 3488
19	3040	2937 2867	1733	1718 1703	1656	1598	1367	1271 1176 1024	-
20	3060	2991 2950 2854	1728	1710	1687	1612	1371	1278 1151 1037	$\nu(\text{p-sub.})$ 831
21	3066	2952 2842	1735	1724	1654	1608	1394	1178 1147 1026	$\nu(\text{p-sub.})$ 842
22	3002	2952 2887	1728	1705	1639	1587	1398	1242 1193 1151	$\nu(\text{O-H})$ 3488

**Figure 1-** FTIR spectrum of compound (11)

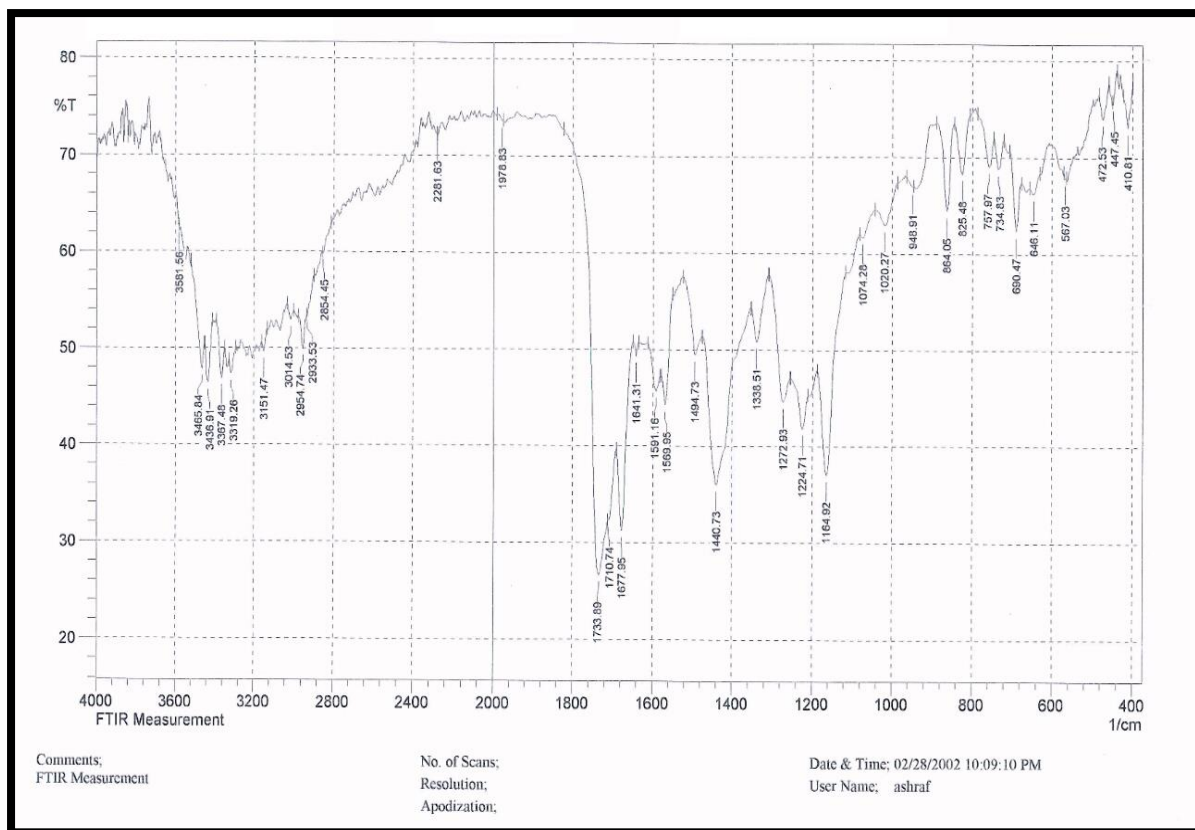


Figure 2- FTIR spectrum of compound (18)

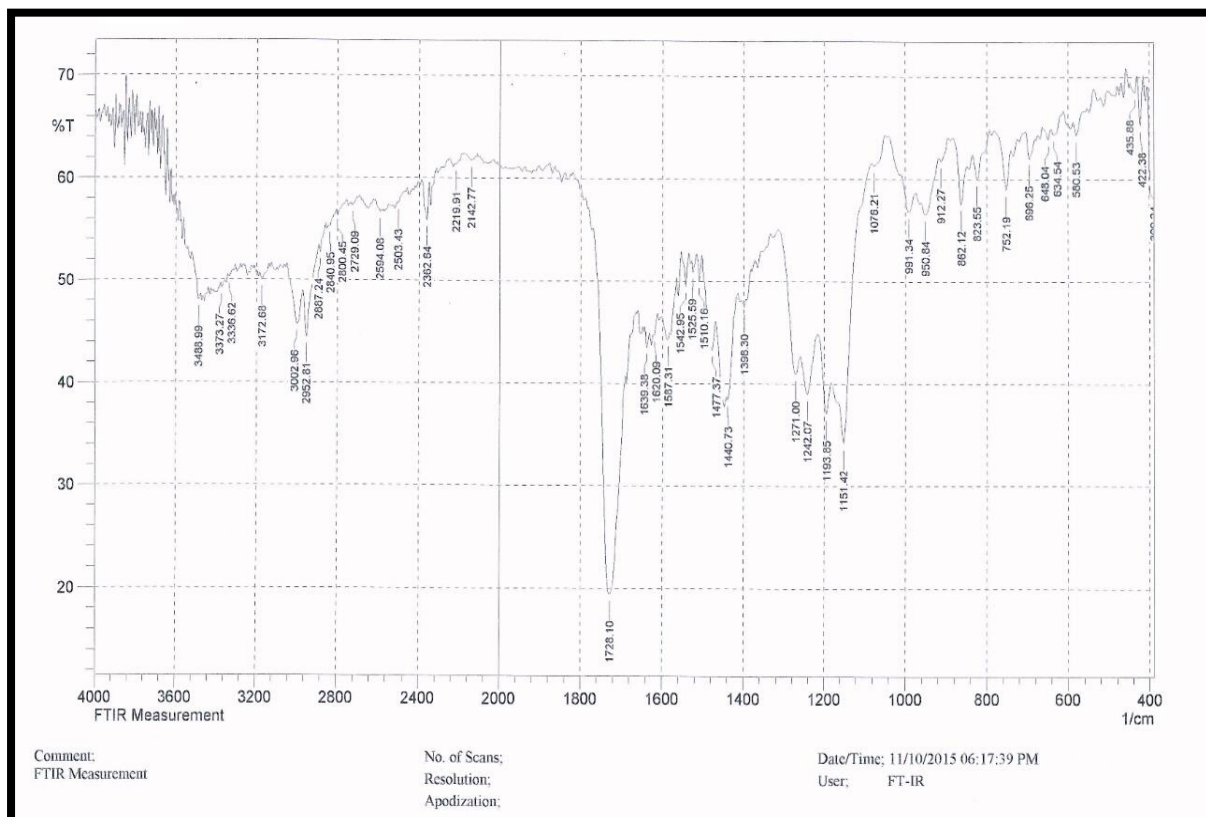


Figure 3- FTIR spectrum of compound (22)

$^1\text{H-NMR}$ spectrum of copolymer (17) showed signals at ($\delta=1.25-1.4$) ppm belong to (CH_2) protons, signal at ($\delta=2$) ppm belong to CH_3 protons and signal at ($\delta=2.8$) ppm belong to aliphatic protons in imide ring and ($-\text{CHCOOCH}_3$) proton. Signals belong to (OCH_3) protons appeared at ($\delta=3.8$) ppm and signals for aromatic protons appeared at ($\delta=7-7.85$) ppm.

$^{13}\text{C-NMR}$ spectrum of copolymer (17) showed signals at ($\delta=22.22$) ppm and ($\delta=27.06$) ppm belong to CH_3 carbon and CH_2 carbon respectively while signal for aliphatic carbons in imide ring and ($-\text{CHCOOCH}_3$) carbon appeared at ($\delta=32.73$) ppm.

The spectrum revealed other clear signals at ($\delta=55.44$) ppm and ($\delta=113.78-126.76$) ppm which belong to (OCH_3) carbon and aromatic carbons, while signals belong to (C=N) and (C=O) carbons appeared at ($\delta=129.59$) and (185.73) ppm respectively [18].

$^1\text{H-NMR}$ spectrum of copolymer (19) showed signals at ($\delta=1.1-1.7$) ppm belong to (CH_2 and CH_3) protons and signal at ($\delta=3.0$) ppm belong to OCH_3 protons and aliphatic protons in imide ring. Signals for aromatic protons appeared at ($\delta=6.2-7.1$) ppm.

$^{13}\text{C-NMR}$ spectrum of copolymer (19) showed signals at ($\delta=21$) ppm and ($\delta=22$) ppm belong to CH_3 carbon and CH_2 carbon respectively. Signals belong to aliphatic carbons in imide ring appeared at ($\delta=23$) ppm while signals belong to ($-\text{C-CO}_2\text{CH}_3$) carbon and (OCH_3) carbon appeared at ($\delta=25$) ppm and ($\delta=54.57$) ppm respectively.

The spectrum showed also signals at ($\delta=112.84-131.29$) ppm belong to aromatic carbons, signals at ($\delta=147.94-148.95$) ppm belong to (C=N) carbons and signals at ($\delta=161.61-162.23$) ppm and ($\delta=162.52-162.69$) ppm belong to (C=O) imide and (C=O) ester carbons.

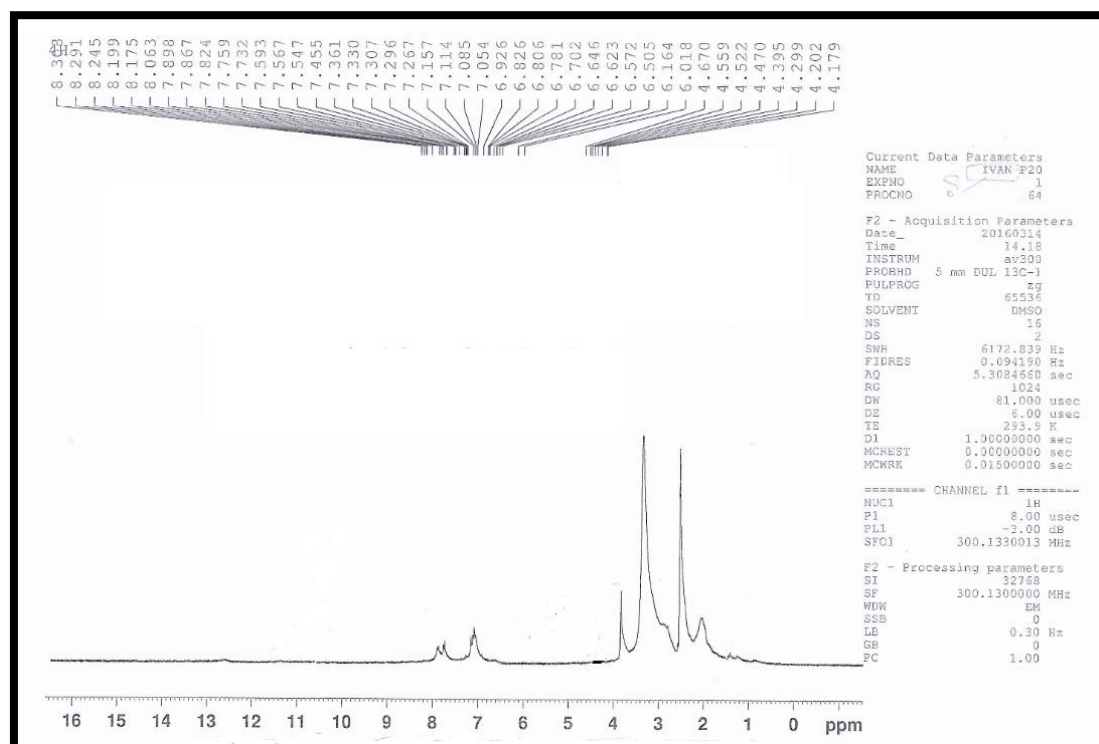
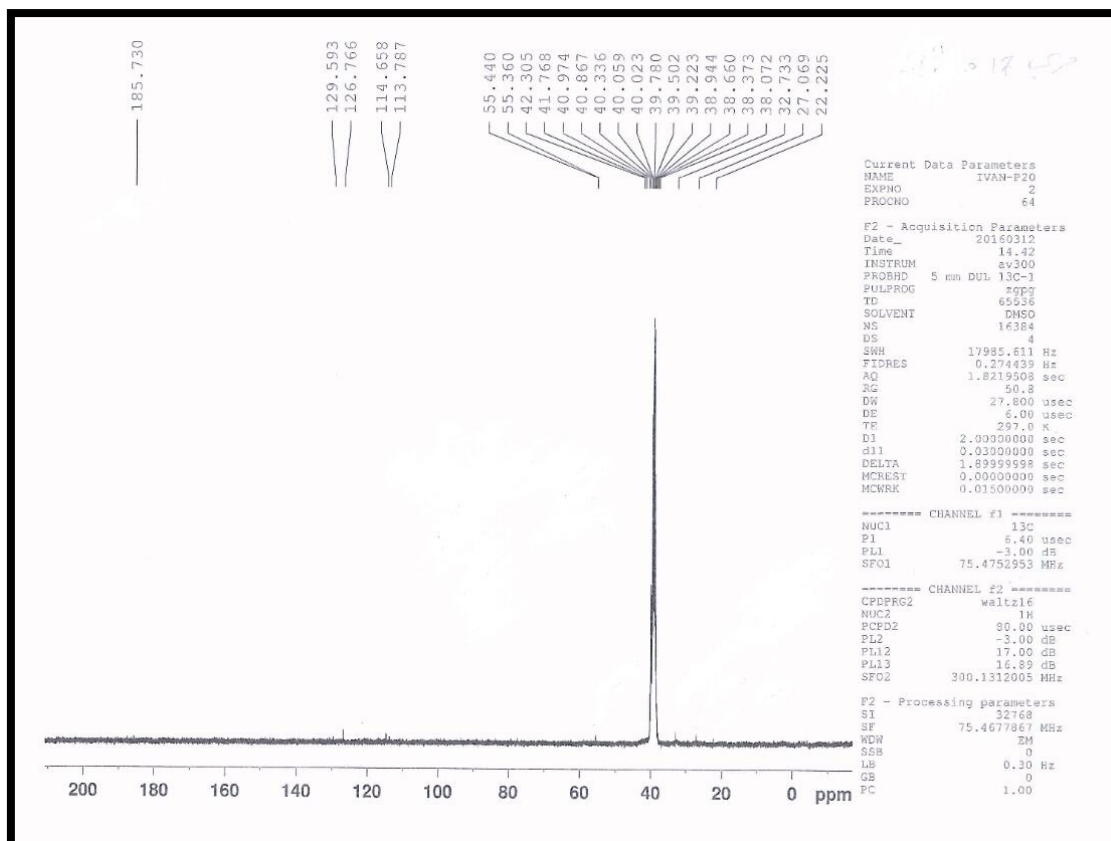
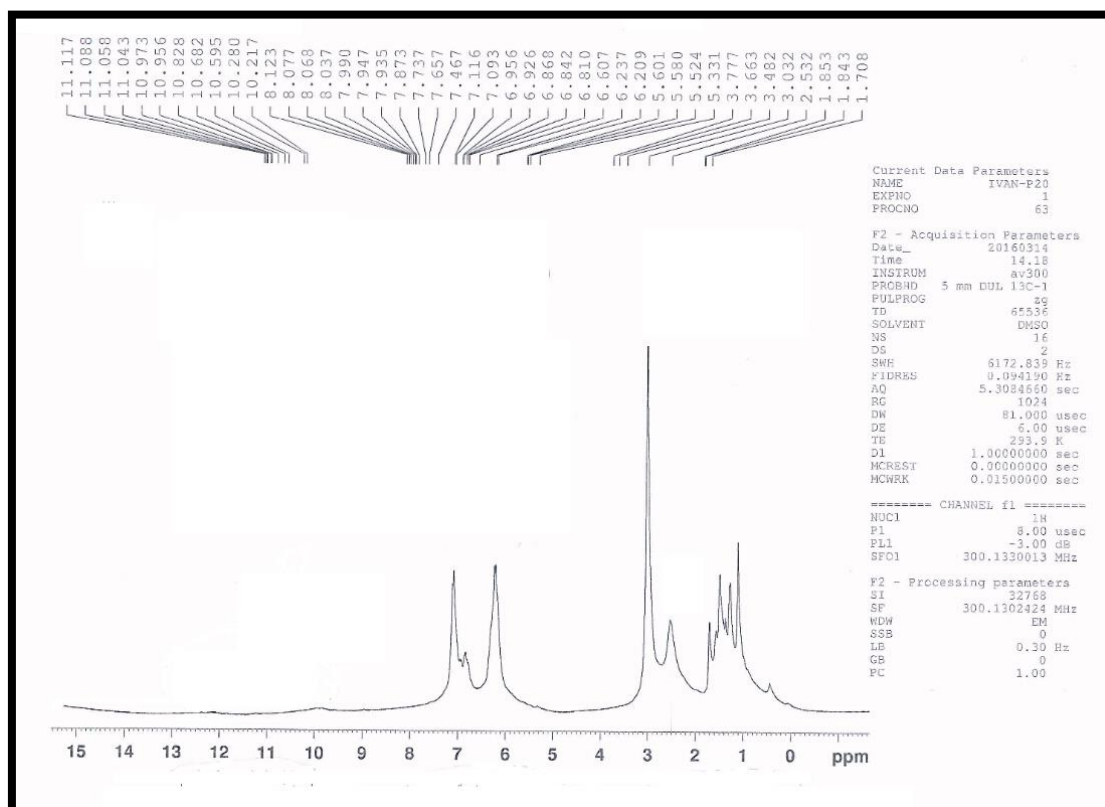


Figure 4- $^1\text{H-NMR}$ spectrum of compound (17)

Figure 5- ^{13}C -NMR spectrum of compound (17)Figure 6- ^1H -NMR spectrum of compound (19)

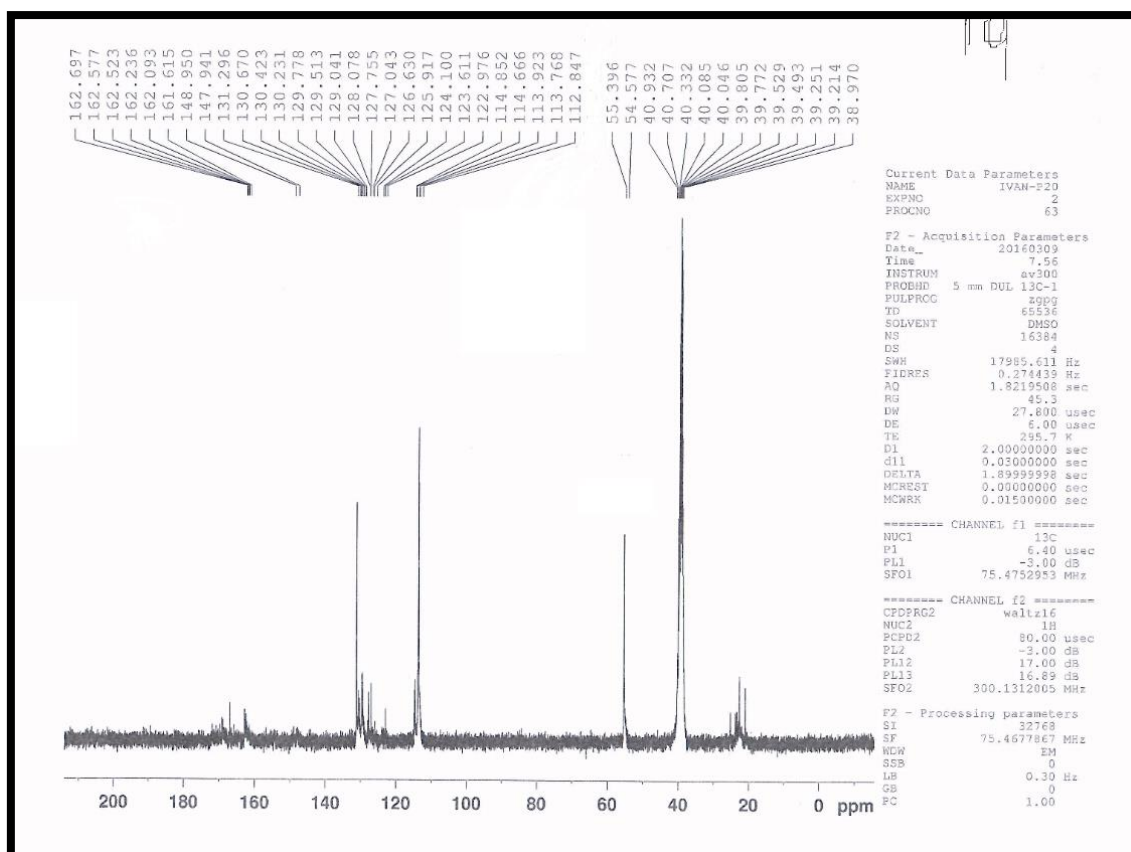


Figure 7- ^{13}C -NMR spectrum of compound (19)

The prepared copolymers in this work showed different physical properties depending on the nature of vinylic monomers used in building their repeating units. Thus the presence of polar cyano group in the repeating units of acrylonitrile and meth acrylonitrile copolymers (11-16) increased intermolecular interaction over all polymeric chains leading to increase crystallinity and this exhibit the polymer high softening points and resistance to solubility in many organic solvents.

On the other hand introducing of methyl acrylate and methyl meth acrylate monomers in copolymerization with oxadiazolyl maleimides exhibit the resulted copolymers softness, flexibility and good solubility in many organic solvents. These physical properties are fitted with the expected ones since incorporation of flexible moieties like ester decrease crystallinity and the total intermolecular interactions and this increase flexibility and solubility [9].

Conclusion

A series of new maleimides bearing oxadiazole moiety were synthesized and introduced in free radical copolymerization with different vinylic monomers producing new copolyimides having different physical properties which may serve different applications.

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