Journal of Al-Nahrain University Vol.11(3), December, 2008, pp.15-30 Science

SYNTHESIS AND CURING OF NOVEL PHENOL-FORMALDEHYDE RESINS CONTAINING PENDANT CITRACONIMIDES

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Abstract

Four novel phenol-formaldehyde resins having pendant citraconimides in their repeating units were synthesized via condensation of formaldehyde with N-(hydroxy phenyl) citraconimides under conditions similar to those used in novolac preparation.

The prepared resins were modified by using two methods the first one involved esterification of phenolic hydroxyl groups via treatment of the resins with different acid chlorides in the presence of triethyl amine producing sixteen new resins while the second method involved free radical polymerization of citracon vinyls of the pendant citraconimides producing four new cross-linked thermally stable polymers.

Both the prepared and the cured resins have new properties which may serve different applications.

Introduction

Phenol-formaldehyde resins are the oldest commercial synthetic polymers, they exhibit excellent electrical and chemical resistance and retention of properties after long term exposure to high temperatures(1-4).

These resins are used in a wide variety of applications including adhesives, surface coatings, binders and molding, therefore not surprisngly a number of synthetic strategies have been carried out to incorporate structural modifications in these resins to obtain new properties serving new applications (5-8).

Experimental

1. Instruments

FTIR spectra were preformed on a shimadzu FTIR 8400 Fourier Transform Infrared spectrophotometer.

^{1}HNMR spectra were recorded on Near Magnetic Resonance Bruker, ultra shield 300 MHz.

UV spectra were recorded on a shimadzu UV-VIS recorder.

Melting points were determined by using Gallen Kamp capillary melting point apparatus and were uncorrected.

Softening points were determined on Thermal microscope Riechert thermover.

Intrinsic viscosities were determined with Automatic viscometer Tafesa Ubbelohde viscometer at 25C^0 using DMSO and chloroform as solvents.

2. Chemicals

All chemicals employed were of analytical reagent grade and were used without further purification.

1-Preparation of N-(hydroxy phenyl) citraconamic acids [1-4]

The titled acids were prepared according to literatures (9) via reaction of citraconic anhydride with different amino phenols.

The prepared acids were purified by recrystallization from dioxane .

Table (1) lists melting points, percent yields and spectral data of the prepared acids.

2-Preparation of N-(hydroxy phenyl) citraconimides [5-8]

The titled imides were prepared by dehydration of the corresponding amic acids according to literature procedures (10) using fusion technique.

The resulted imides were purified by recrystallization from cyclohexane.

Table (2) lists melting points, percent yields and spectral data of the prepared imides.

3-Preparation of N-citraconimidyl phenol-formaldehyde resins [9-12]

2.03g (0.01mole)of N-(hydroxy phenyl) citraconimide, (0.01mole) of formalin 37% and (10 ml) DMF were placed in three neck round bottomed flask fitted with a reflux condenser, thermometer and dropping funnel.

The dropping funnel was charged with (0.1ml) of conc. H_2SO_4 and (0.5 ml) of distilled water then this solution was added drope wise with stirring and the mixture was refluxed at 110 C^0 for 3 hrs.

The evolved water and solvent were distilled off under reduced pressure and the polymeric residue was dissolved in DMF then filtered and the filtrate was added to excess water to precipitate the polymer.

The resulted polymer was filtered, washed with petroleum ether and dried in an oven at (65-70) C^0 for 24 hrs.

Table (3) lists some physical properties, U.V data and major IR absorptions of the prepared resins.

Curing of the prepared phenol-formaldehyde resins

Curing of the prepared phenolic resins was performed by two methods as described below:

A-Curing via esterification of substituted hydroxyl groups in the prepared resins

Curing of the prepared resins by esterification involved the following preparations:

4-Esterification of the prepared phenolic resins to the corresponding poly [N-(benzoyloxy phenyl - formaldehyde) citraconimides][13-16]

Esterification of phenolic resins was performed according to literature procedures(11) with some modifications:

(0.01 mole) of the prepared citraconimidyl phenolic resins, (20 ml) of DMF and (0.01 mole) of Et_3N were placed in a suitable round bottomed flask fitted with pressure-equalized funnel which was charged with (0.01 mole) of benzoyl chloride.

Benzoyl chloride was added dropwise to the mixture with stirring and cooling to (5 C^0) then stirring was continued for another six hrs at room temperature.

The mixture was filtered then the filtrate was evaporated under reduced pressure and the residue was extracted with water and chloroform for three times.

The organic layer was dried and the solvent was evaporated to yield a thick oil which was purified by passing its chloroform solution through a florisil column.

Table (4) lists physical properties, U.V absorptions and major IR absorptions of the cured resins [13-16].

5-Esterification of the prepared phenolic resins to the corresponding poly [N-(methacryloyloxy phenyl-formaldehyde) citraconimides] [17-20]

The titled compounds were prepared by following the same procedure used in the preparation of the resins [13-16] except using of methacryloyl chloride instead of benzoyl chloride.

Table (5) lists physical properties, U.V absorptions and major IR absorptions of the cured resins [17-20].

6-Esterification of the prepared phenolic resins to the corresponding poly [N-(acryloyloxy phenyl - formaldehyde) citraconimides] [21-24]

The titled compounds were prepared by following the same procedure used in the preparation of the resins [13-16] except using of acryloyl chloride instead of benzoyl chloride

Table (6) lists physical properties, U.V absorptions and major IR absorptions of the cured resins [21-24].

7-Esterification of the prepared phenolic resins to the corresponding poly [N-(cinnamoyloxy phenyl - formaldehyde) citraconimides] [25-28]

The titled compounds were prepared by following the same procedure used in the preparation of the resins [13-16] except using of cinnamoyl chloride instead of benzoyl chloride

Table (7) lists physical properties, U.V absorptions and major IR absorptions of the cured resins [25-28].

B-Curing of the prepared resins via free radical polymerization [29-32]

Free radical polymerization of vinylic bonds in citraconimide rings was performed according to literatures(12) with some modifications.

In a suitable polymerization bottle (0.01 mole) of the prepared phenolic resin [9-12] was dissolved in (8 ml) of DMF then (0.0002 g) of benzoyl peroxide was added as initiator.

The bottle was flashed with N_2 gas, stoppered tightly and heated in an oil bath at (110-115) C^0 for 3 hrs.

The resulted mixture was added to (50 ml) of cold distilled water then the precipitated polymer was filtered, washed with petroleum ether and dried.

Table (8) lists physical properties and spectral data of the cured resins [29-32].

Results and Discussion

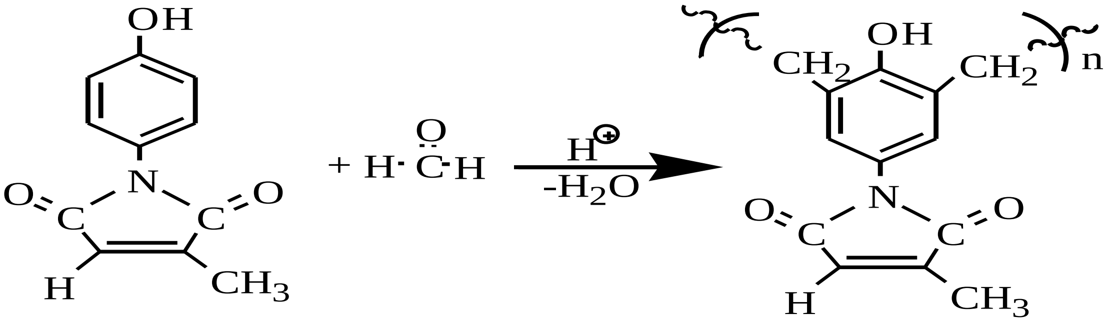
The aim of the present investigation is to synthesize new phenol formaldehyde resins having new properties by following new strategy.

To perform this target many steps have to be done, the first step involved reaction of ortho, meta and para amino phenols with citraconic anhydride producing N-(hydroxyl phenyl) citraconamic acids.

Dehydration of the prepared acids was achieved in the second step by using fusion technique to produce the corresponding N-(hydroxy phenyl) citraconimides.

Our new strategy used in this work in building new phenolic resins based on introducing of new phenolic moiety represented by phenolic ring attached to citraconimide ring in condensation reaction with formaldehyde.

Condensation was performed under acidic conditions similar to those used in novolac preparation as shown in the following equation(13)



Incorporation of citraconimide groups in the structural units of the prepared resins played a vital role in affecting physical properties of the resulted resins since insertion of citraconimidyl units into some polymer chains was greatly improved heat resistance, solvent resistance and chemical stability of these polymers (14).

The obtained phenolic resins were red to brown solids and the presence of pendant citraconimide rings in their chains exhibit them good resistance to solubility in most organic solvents and high softening points.

Intrinsic viscosities of these resins were in the range (0.55-0.61) dLl/g.

Structures of the prepared phenolic resins were confirmed by FTIR, ^{1}HNMR and U.V spectra.

FTIR spectra of the prepared resins [9-12] revealed many characteristic absorption bands at (3350-3420) cm^{-1}, (1650-1710) cm^{-1}, (1600-1655) cm^{-1}, (1375-1390) cm^{-1} and (2900-2985) cm^{-1} which were attributed to υ(O-H) phenolic, υ(C=O) imide, υ(C=C) vinylic, υ (C-N) and υ (C-H) aliphatic respectively(15).

^{1}HNMR spectra of the prepared resins [9-12] revealed many characteristic signals including signals at δ(1.25-1.9)ppm for CH_3 protons, δ(3.7-3.8)ppm for methylene protons, δ(6.7) ppm for vinylic proton, δ(6.9-7.1)ppm for aromatic protons and δ(9.8-9.9)ppm for phenolic OH proton.

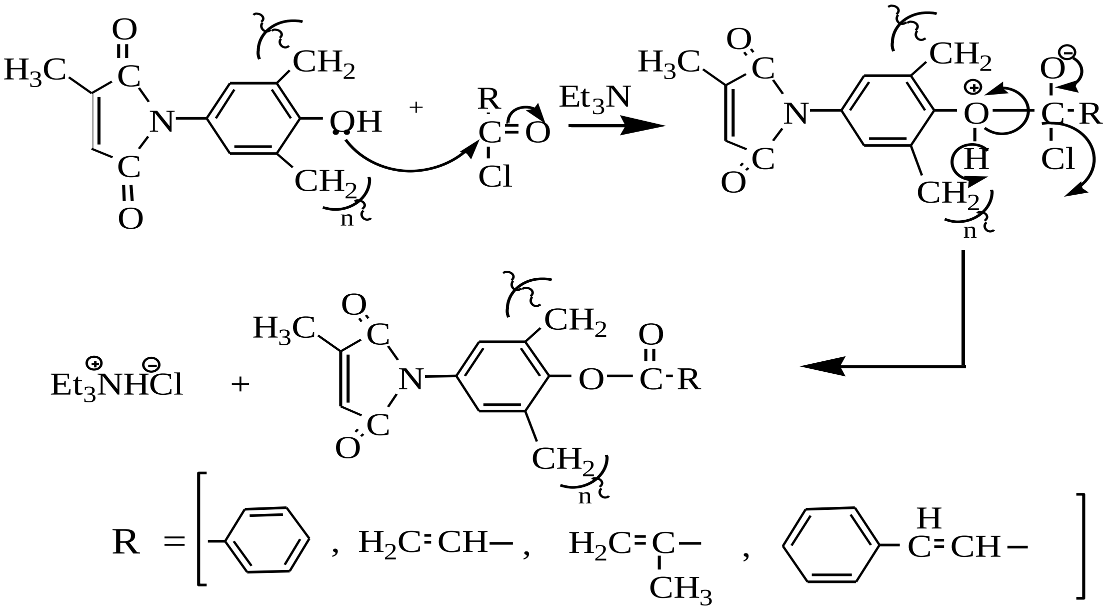
On the other hand U.V spectra of these resins showed bands at λmax (288-294) nm due to $\pi \rightarrow \pi^*$ transitions and $n \rightarrow \pi^*$ in the conjugated enone system of citracon ring and attached phenolic ring (15,16) .

The second part of this work involved curing of the prepared resins.

Curing was performed by two ways the first one involved esterification of phenolic hydroxyl groups in the prepared resins.

Esterification was carried out by treatment of the prepared resins with different acid chlorides including acryloyl, benzoyl, methacryloyl and cinnamoyl chlorides in the presence of triethyl amine.

Mechanism of esterification involved nucleophilic attack of phenolic hydroxyl groups on electron deficient carbon of carbonyl groups in acid chlorides as illustrated by scheme (1) (17).



Scheme (1).

It was found that esterification method was quiet convenient to incorporate structural modification in the prepared resins [9-12] since the modified resins have quiet different physical properties than those of the starting resins (8).

The resulted modified resins were yellow to brown oils, soluble in all organic solvents and have low intrinsic viscosities in the range (0.13-0.37) dL/g .

These physical properties were agreed with the expected ones due to the presence of (C-O-C) ester bonds in the incorporated ester moiety which exhibit the modified polymers softness and flexibility and this lead to decrease their softening points and resistance to solubility which inturn made these resins suitable for certain applications

like varnishes, additives for lubricating oils and adhesives (3,6).

FTIR ^{1}HNMR and U.V spectra were used to confirm the structures of the cured resins [13-28], moreover these cured resins gave positive results in specific test of esters.

FTIR spectra of the cured resins [13-28] revealed two characteristic clear absorption bands in the regions (1730-1790) cm^{-1} and (1175-1232) cm^{-1} which were attributed to υ(C=O) ester and υ (C-O-C) ester respectively, thus the presence of these two absorption bands and disappearance of υ (O-H) phenolic absorption band were good proofs for the success of esterification reaction.

Other absorption bands appeared at (1647-1740) cm^{-1}, (1571-1650) cm^{-1} , (1300-1396) cm^{-1} and (2870-2980) cm^{-1} due to υ (C=O) imide, υ (C=C) vinylic, υ (C-N) and υ(C-H) aliphatic respectively (15,16,17) .

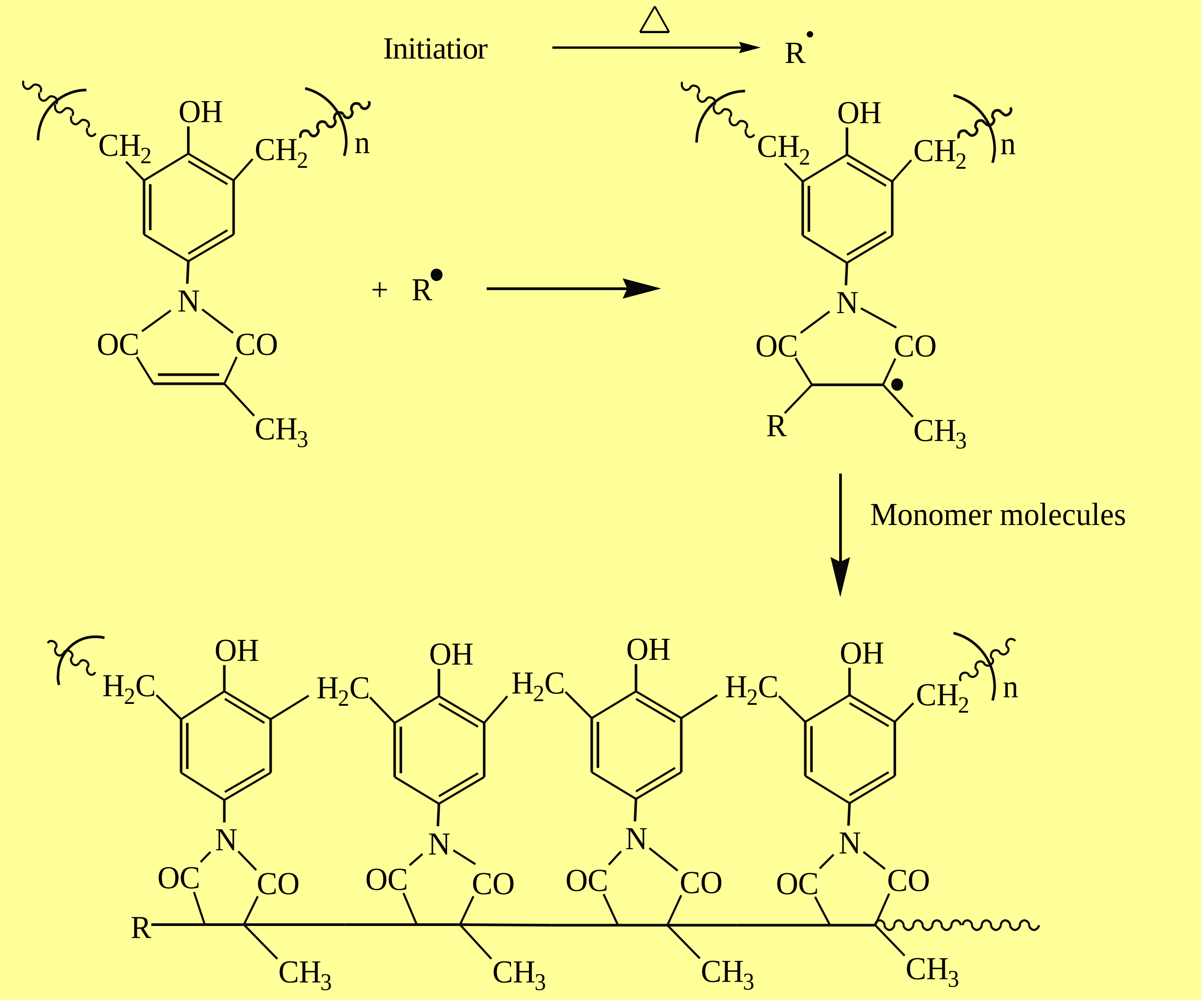
^{1}HNMR spectra of the cured resins showed disappearance of the signal belong to phenolic OH proton and this is a clear proof which confirmed the success of estrification reaction. At the same time these NMR spectra revealed many characteristic signals including signals at δ(1.3-1.4) ppm for CH_3protons, δ(3.1-3.8)ppm for methylene protons, δ(6.2-7.1)ppm for vinylic protons and signals at δ(7.3-8.1)ppm for aromatic protons.

On the other hand U.V spectra of these resins [13-28] showed bands at λ max (300-348) nm due to $\pi \rightarrow \pi^*$ transitions $n \rightarrow \pi^*$ and in vinyl group which was conjugated with two carbonyl groups in citraconimide ring and in phenyl ring which was conjugated with attached benzoyloxy, acryloyloxy, methacryloxy and cinnamoyloxy groups.

Since the prepared phenolic resins [9-12] have another reactive site represented by vinylic (C=C) bonds in citracon rings we can incorporate structural modification in these resins via free radical polymerization of these vinylic bonds(12) .

Polymerization was proceeded success-fully by using benzoyl peroxide as nitiatori and DMF as solvent to produce four cross-linked thermally stable polymers [29-32].

The mechanism of polymeriz-ation reaction was expressed by equations in Scheme (2) (17).

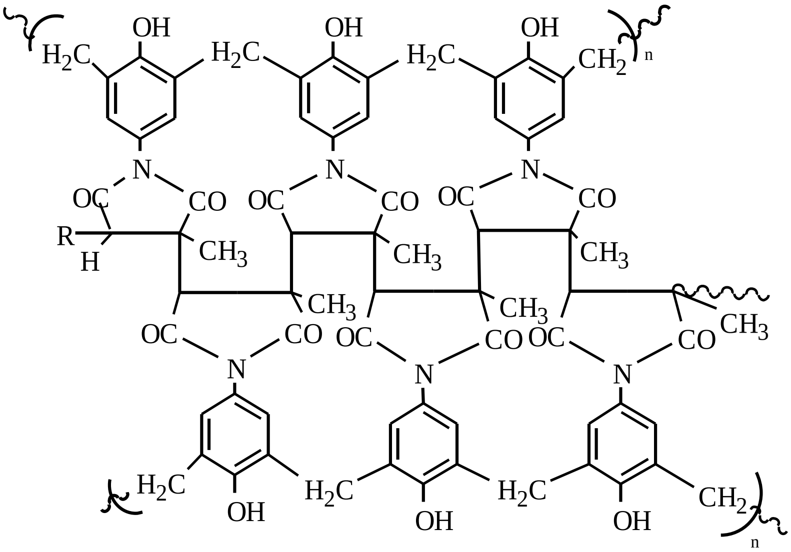


Scheme (2).

The resulted cured polymers were brown to black solids which stay un soften at (300 C^0) and some of them were insoluble in all organic solvents while others were soluble only in DMSO and their intrinsic viscosities were in the range (0.96-1.2) dL/g.

It was noteworthy that the presence of polyimide chains in the cured resins [29-32] have a noticeable effect on physical properties of these resins thus the key reasons for insolubility, thermal stability and non melting character of the cured resins [29-32] were the lack of flexibility and strong interchain interactions offered by polyimide chains (18-21).

Also curing via polymerization caused cross-linking between polymeric chains which inturn lead finally to thermal stable polymers. Cross-linking between polymeric chains may be illustrated by the following suggested structure(17) .



FTIR spectra of the cured resins [29-32] showed disappearance of absorption band belong to υ(C=C) vinylic due to saturation during polymerization.

Success of polymerization of citracon vinyls was confirmed also by $KMnO_4$ and Br_2/CCl_4 tests which gave negative results proving the saturation of vinylic bonds during polymerization.

Physical properties,UV absorptions and major IR absorptions for all the prepared compounds in this work are listed in Tables (1-8) while ^{1}HNMR spectral data for some of the prepared compounds are listed in Table (IX).

FTIR and ^{1}HNMR spectra of some of the prepared compounds are shown in Figs. (1-10).

As a final conclusion the present investigation provides a suitable strategies for building new phenolic resins via choosing of new phenolic moiety to condense with formaldehyde and using of esterification and chain polymerization to incorporate structural modifications in the prepared resins producing twenty new phenolic resins having new properties which fitted with different applications.

Table (1)
Physical properties and spectral data of citraconamic acids.

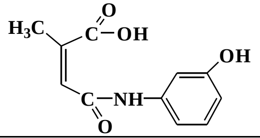
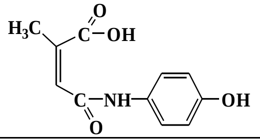
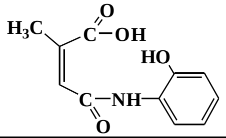
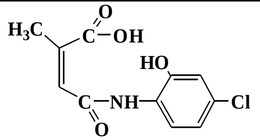
Comp. No.	Compound structure	Color	Yield %	Melting Point C^0	U.V λmax nm	Major IR absorptions cm^{-1}				
						υ O-H phenolic	υ O-H carboxylic	υ N-H Amide	υ C=O carboxylic	υ C=O Amide
1		Yellow	92	154-155	282	3362	3227	3317	1701	1630
2		Yellow	89	157-158	291	3540	3230	3325	1710	1625
3		Yellow	93	150-152	297	3390	3091	3356	1701	1626
4		Yellow	86	159-160	270	3300	3080	3175	1710	1625

Table (2)
Physical properties and spectral data of citraconimides.

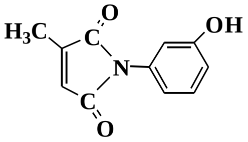
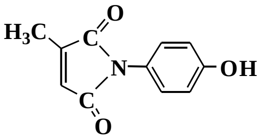
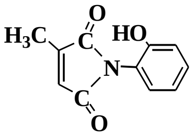
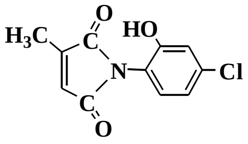
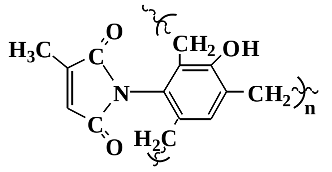
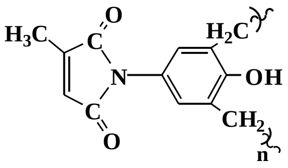
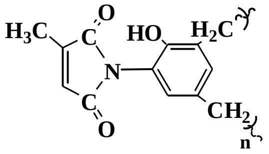
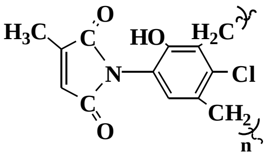
Comp. No.	Compound structure	Color	Yield %	Melting Point C^0	U.V λmax nm	Major IR absorptions cm^{-1}			
						υ O-H phenolic	υ C=O imide	υ C=C Vinylic	υ C-N
5		Yellow	86	148-150	237	3325	1700	1650	1380
6		Yellow	82	166-167	240	3360	1690	1641	1377
7		Red	80	134-136	235	3398	1700	1641	1348
8		Red	85	170-172	240	3370	1710	1650	1380

Table (3)
Physical properties and spectral data of poly[N-(phenol-formaldehyde) citraconimides].

Comp. No.	**Polymer**	**Color**	**S.P.°C**	**[η] dL/gm**	**Conv. %**	**U.V λmax nm**	**Major IR absorptions cm^{-1}**			
							υ phenolic	**υC=O imide**	**υC=C Vinylic**	**υC-N**
9		Red	>300	*	91	*	3369	1710	1655	1388
10		Brown	230	0.59	81	288	3350	1700	1630	1390
11		Pale brown	>300	0.55	79	294	3400	1650	1600	1390
12		Brown	255	0.61	83	291	3420	1701	1610	1375

** Insoluble in all organic solvents*

Table (4)
Physical properties and spectral data of poly [N-benzoyloxy phenyl-formaldehyde) citraconimides].

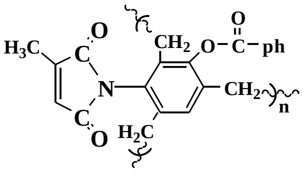
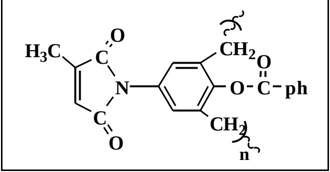
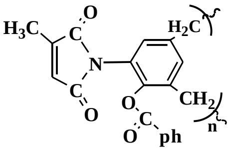
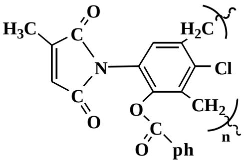
Comp. No.	**Polymer**	**Color**	**S.P.°C**	**[η] dL/gm**	**Conv. %**	**U.V λmax nm**	**Major IR absorptions cm^{-1}**			
							υC=O Ester	**υC=O Imide**	**υC-O-C Ester**	**υC-N**
13		Brown	Oil	0.32	76	309	1790	1710	1210	1380
14		Yellow	Oil	0.13	72	300	1790	1710	1210	1375
15		Deep brown	Oil	0.18	70	324	1790	1700	1200	1360
16		Brown	Oil	0.35	72	305	1785	1687	1209	1390

Table (5)
Physical properties and spectral data of poly [N- methacryloyloxy phenyl-formaldehyde) citraconimides].

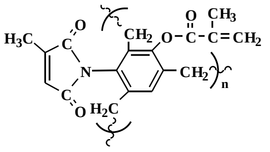
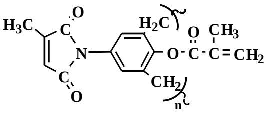
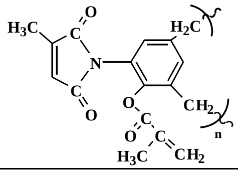
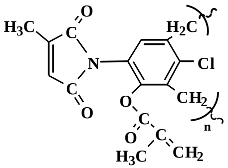
Comp. No.	Polymer	Color	S.P.°C	[η] dL/gm	Conv. %	U.V λmax nm	Major IR absorptions cm^{-1}				
							υC=O Ester	υC=O Imide	υC=C Vinylic	υC-O-C Ester	υC-N
17		Brown	Oil	0.19	72	333	1760	1740	1640	1180	1390
18		Brown	Oil	0.24	76	340	1755	1710	1645	1190	1390
19		Pale brown	Oil	0.21	71	338	1735	1720	1650	1175	1380
20		Pale brown	Oil	0.18	64	332	1732	1647	1571	1190	1373

Table (6)
Physical properties and spectral data of poly [N- Acryloyloxy phenyl-formaldehyde) citraconimides].

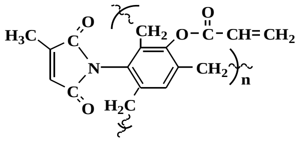
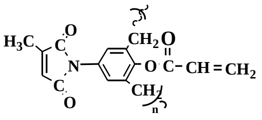
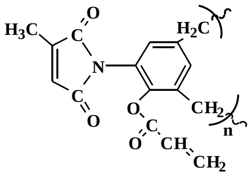
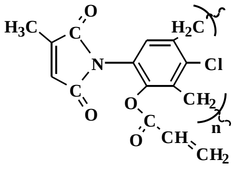
Comp. No.	Polymer	Color	S.P.°C	[η] dL/gm	Conv. %	U.V λmax nm	Major IR absorptions cm^{-1}				
							υC=O Ester	υC=O Imide	υC=C vinylic	υC-O-C Ester	υC-N
21		Yellow	Oil	0.33	73	330	1745	1720	1640	1200	1360
22		Brown	Oil	0.35	72	336	1740	1650	1600	1210	1380
23		Pale brown	Oil	0.33	70	334	1730	1650	1600	1200	1390
24		Brown	Oil	0.27	66	334	1730	1650	1597	1230	1396

Table (7)
Physical properties and spectral data of poly [N-cinnamoyloxy phenyl-formaldehyde) citraconimides].

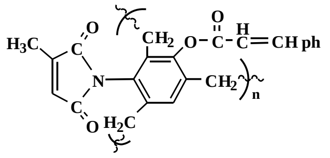
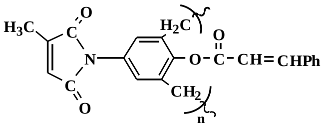
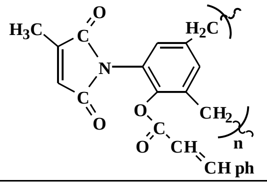
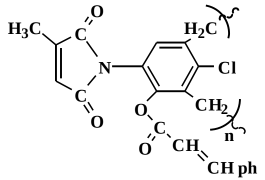
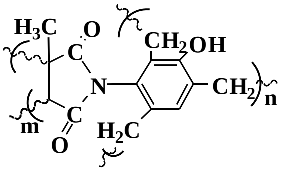
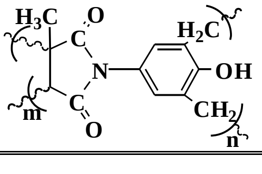
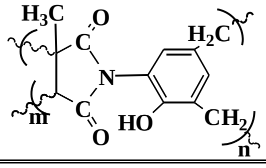
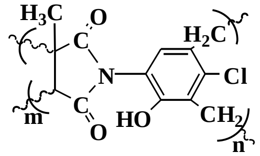
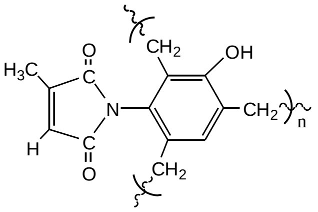
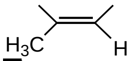
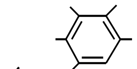
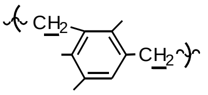
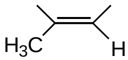
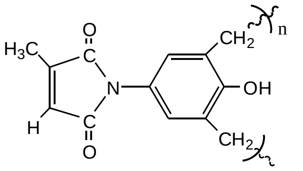
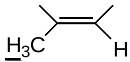
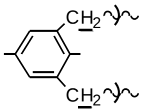
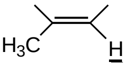
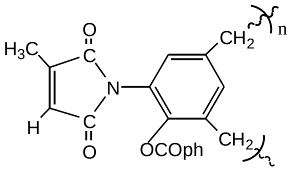
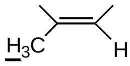
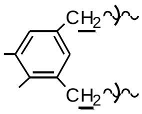
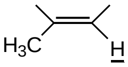
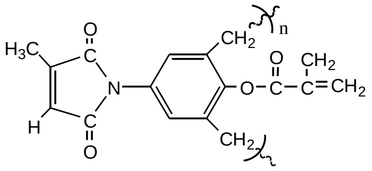
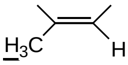
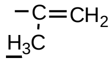
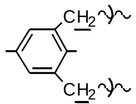
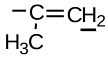
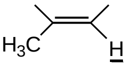
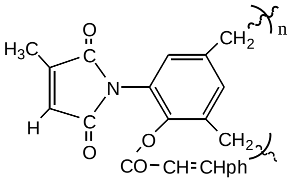
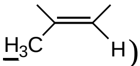
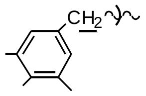
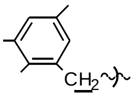
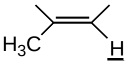
Comp. No.	Polymer	Color	S.P. °C	[η] dL/gm	Conv . %	U.V λmax nm	Major IR absorptions cm^{-1}				
							υC=O Ester	υC=O Imide	υC=C Vinylic	υC-O-C Ester	υC-N
25		Pale brown	Oil	0.23	80	345	1760	1700	1645	1200	1300
26		Yellow	Oil	0.37	77	348	1760	1710	1625	1205	1300
27		Brown	Oil	0.22	83	338	1770	1700	1640	1200	1300
28		Pale brown	Oil	0.2	78	338	1763	1700	1650	1232	1388

Table (8)
Physical properties and spectral data of the cured resins [29-32].

Comp. No.	Polymer	Color	S.P. °C	[η] dL/gm	Conv. %	U.V λmax nm	Major IR absorptions cm^{-1}		
							υ O-H Phenolic	υC=O Imide	υC-N
29		Brown	>300	* ——	82	——	3375	1700	1380
30		Black	>300	* ——	73	——	3400	1700	1390
31		Brown	>300	1.2	76	345	3407	1701	1388
32		Brown	>300	0.96	78	342	3385	1701	1388

** Insoluble in all organic solvents.*

Table (9)
^{1}HNMR chemical shifts for some of the prepared compounds.

Comp. No.	Compounds structure	Chemical shift ppm
9		δ1.25 (3H, ), δ2.2 (2H, ), δ3.7 (4H, ), δ6.7 (1H, ), δ7.1 (1H aromatic), δ9.9 (1H, OH phenolic)
10		δ1.9(3H, ), δ3.8(4H, ), δ6.7 (1H, ), δ(6.9, 7.1) (2H aromatic), δ9.7 (1H, OH phenolic)
15		δ1.4(3H, ), δ3.2(4H, ), δ7.1 (1H, ), δ(7.5,7.6,8.1) (7H, aromatic)
18		δ1.4(6H, , ),δ3.1(4H, ), δ3.8(2H, ), δ6.2(1H, ), δ7.3(2H, aromatic)
27		δ1.3(3H, ), δ3.1(2H, ), δ3.5(2H, ), δ4.2(1H, −CH=CHph) δ6.4(1H, −OCO−CH=CHph), δ6.8(1H, ), δ(7.3, 7.5, 7.7) (8H, aromatic)

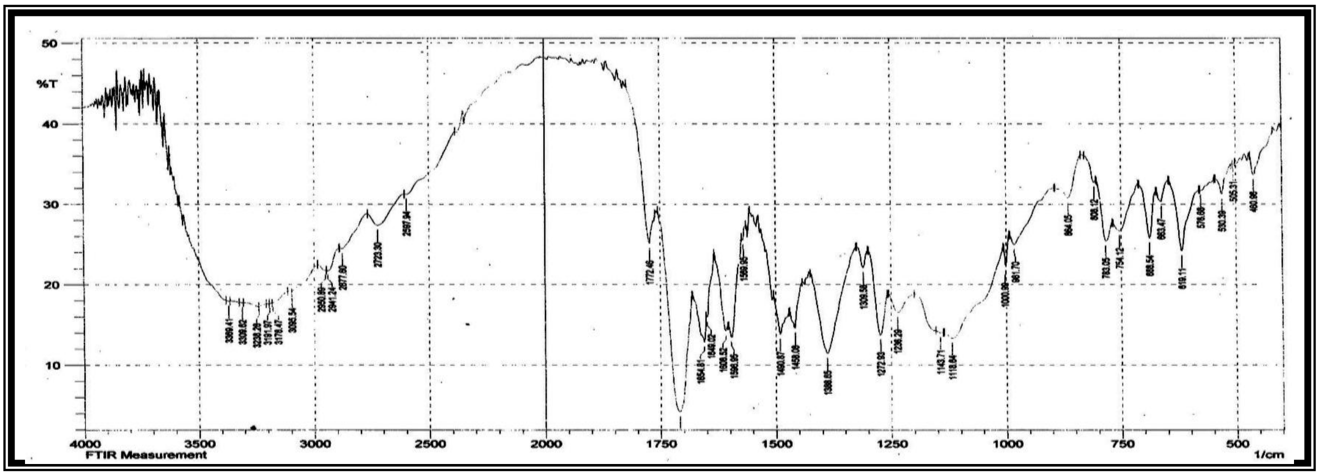


Fig. (1) : FTIR spectrum for compound No. [9].

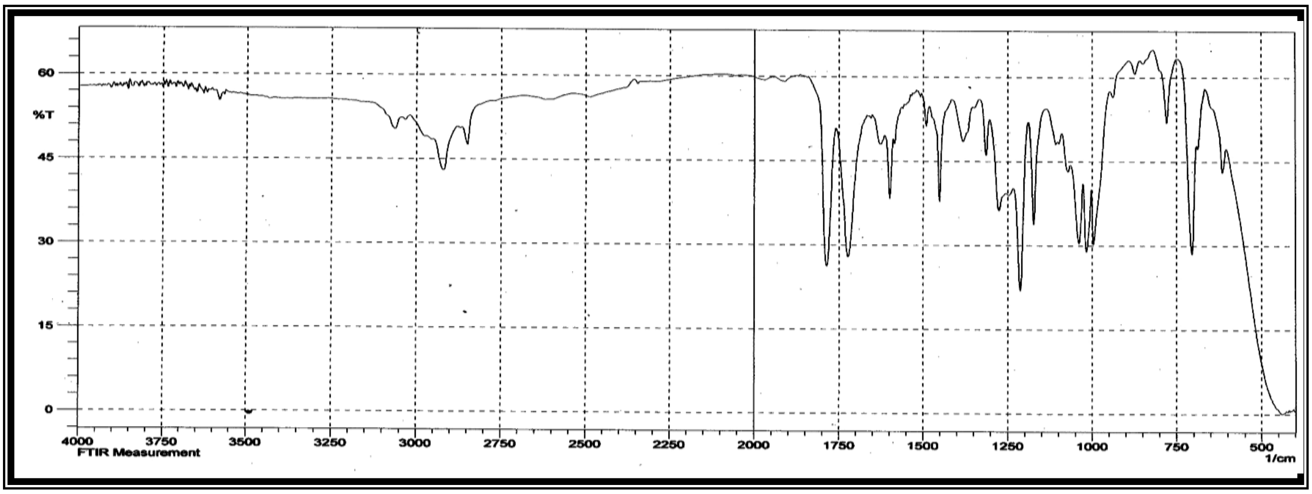


Fig. (2) : FTIR spectrum for compound No. [13].

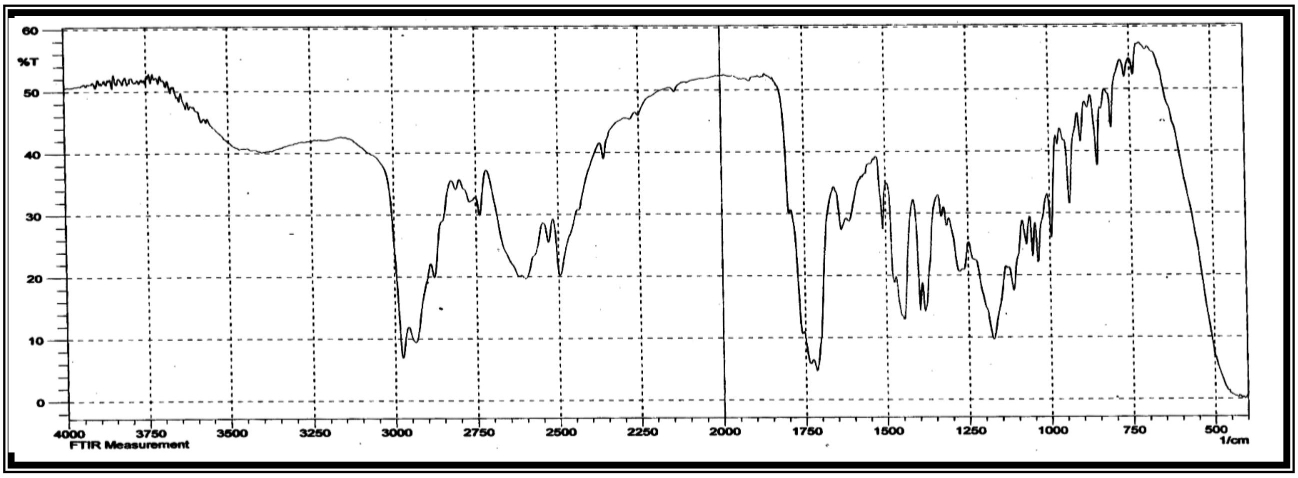


Fig. (3) : FTIR spectrum for compound No. [18].

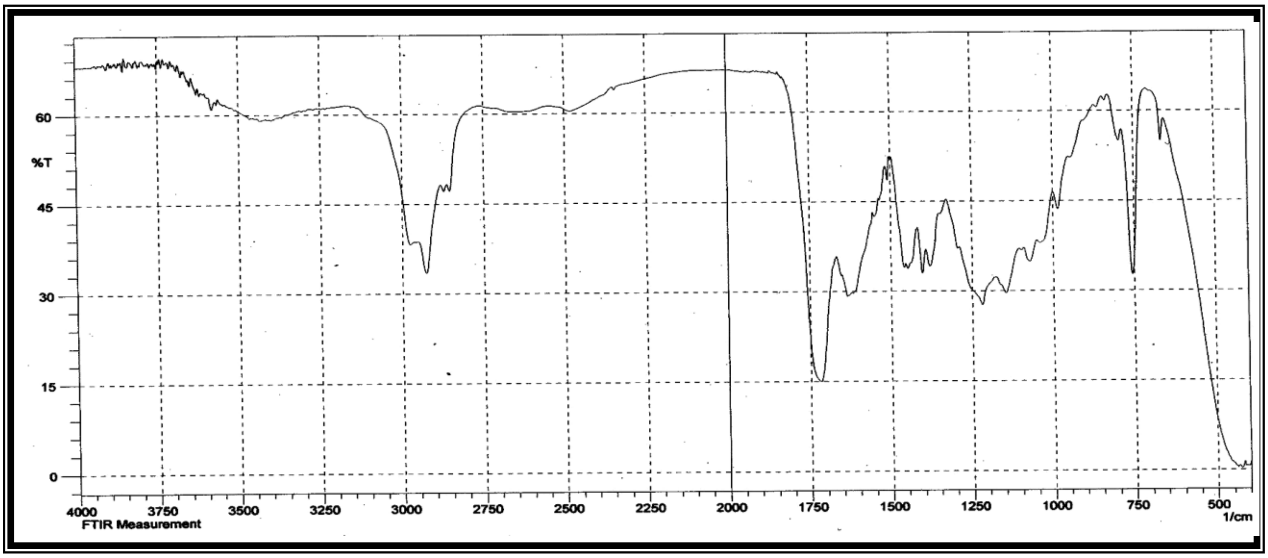


Fig. (4) : FTIR spectrum for compound No. [22].

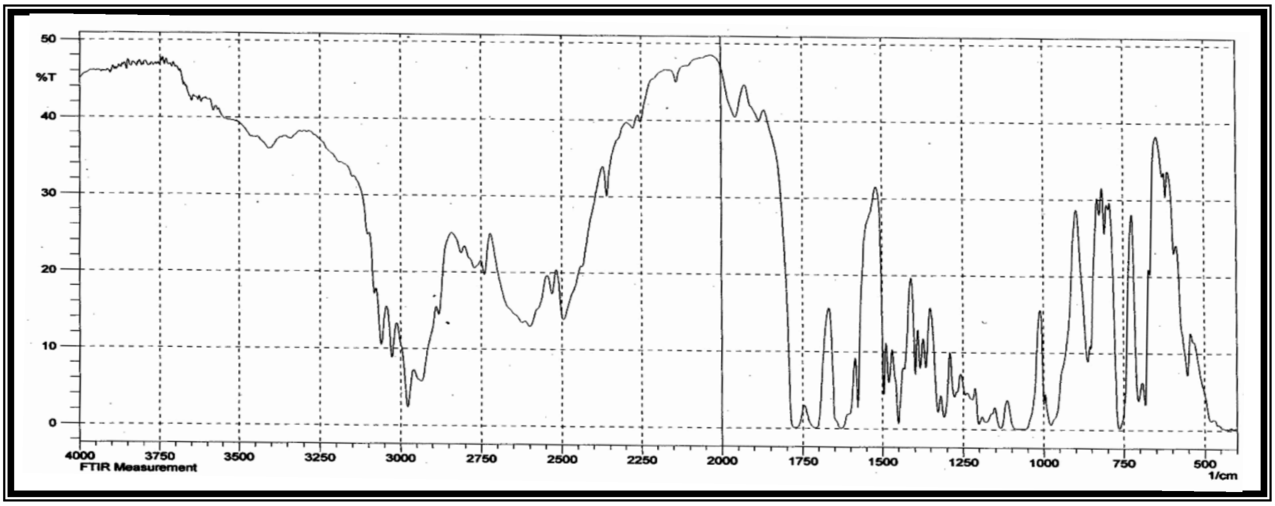


Fig. (5) : FTIR spectrum for compound No. [27].

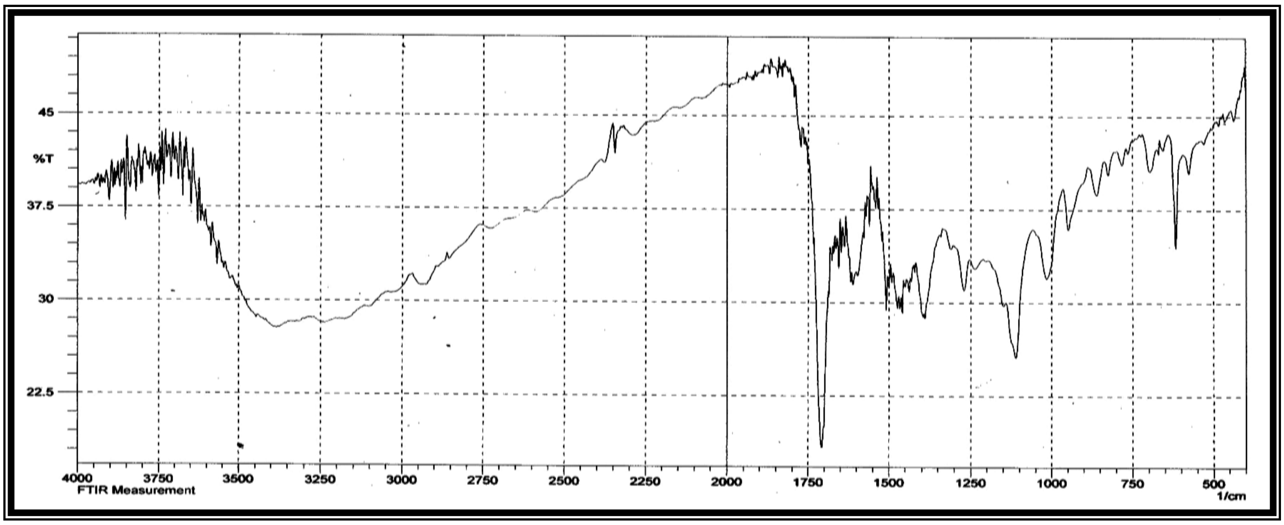
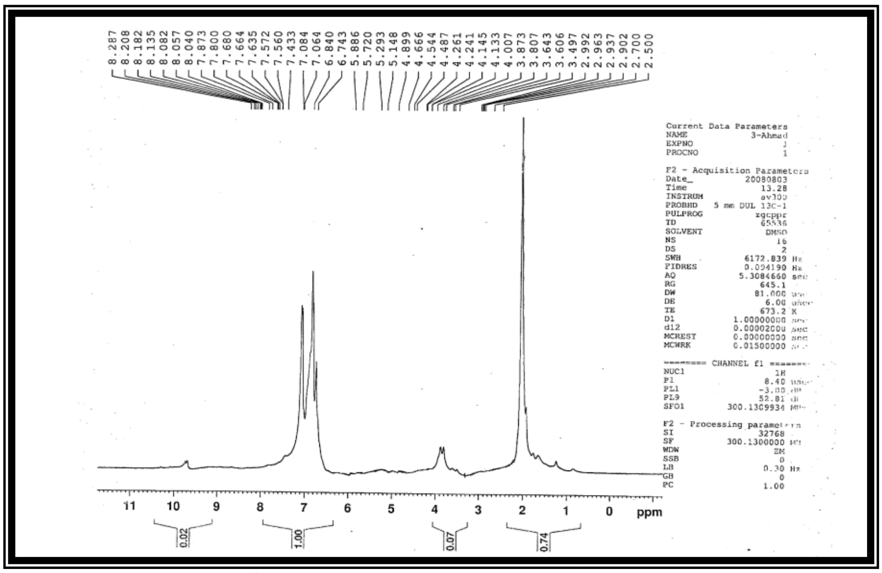


Fig. (6) : FTIR spectrum for compound No. [29].



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EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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Time                13.28
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PULPROG            zgcppr
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ              5.3084660 sec
RG                  645.1
DW                 81.000 usec
DE                   6.00 usec
TE                  673.2 K
D1             1.00000000 sec
d12            0.00002000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.40 usec
PL1                 -3.00 dB
PL9                 52.81 dB
SFO1          300.1309934 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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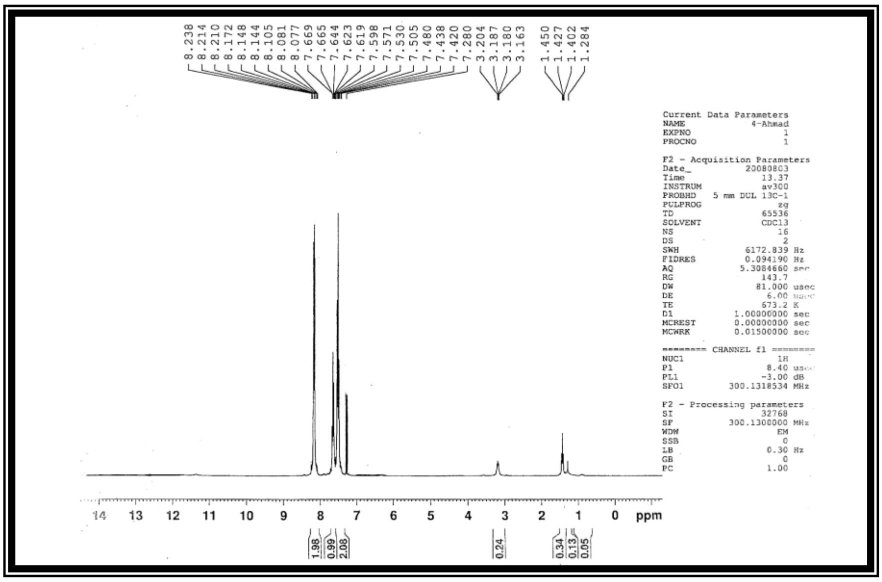
Fig.(7):^{1}HNMR spectrum of compound No.[9].



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Current Data Parameters
NAME              4-Ahmad
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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PULPROG                zg
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ              5.3084660 sec
RG                  143.7
DW                 81.000 usec
DE                   6.00 usec
TE                  673.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.40 usec
PL1                 -3.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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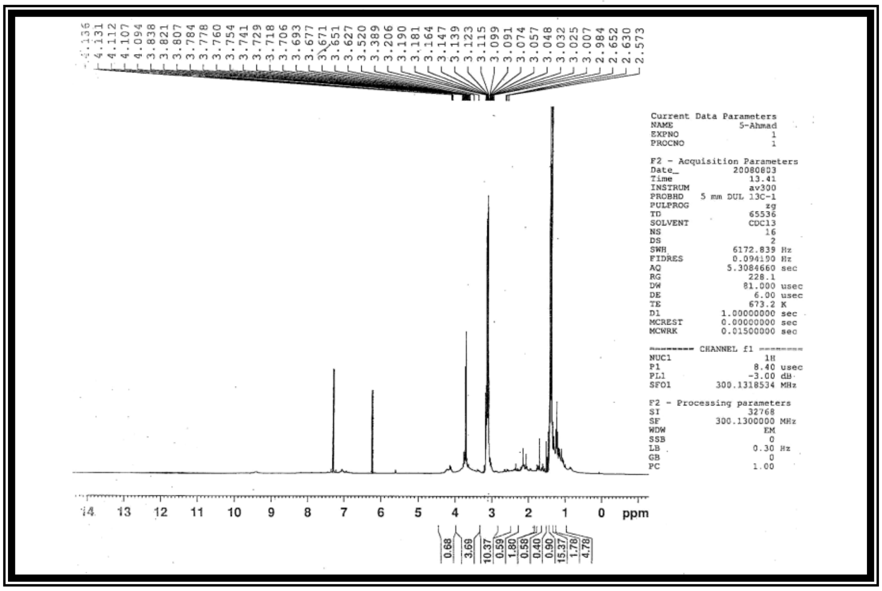
Fig.(8) : ^{1}HNMR spectrum of compound No.[15].



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Current Data Parameters
NAME              5-Ahmad
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
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PULPROG                zg
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SOLVENT             CDC13
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ              5.3084660 sec
RG                  228.1
DW                 81.000 usec
DE                   6.00 usec
TE                  673.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.40 usec
PL1                 -3.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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Fig.(9): ^{1}HNMR spectrum of compound No.[18].

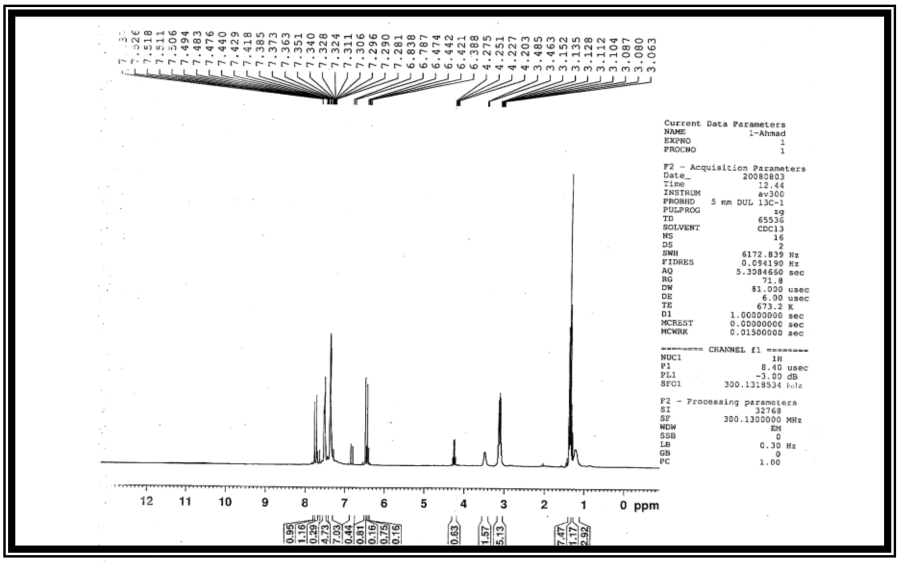

Fig.(10): ^{1}HNMR spectrum of compound No.[27].

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الخلاصة

تضمن البحث تحضير أربعة راتنجات فينولية جديدة معوضة بمجاميع متدلية من الستراكون ايمايدات في وحداتها المتكررة وذلك من خلال تكاثف الفورمالديهايد مع مركبات N – (هيدروكسي فنيل) ستراكون ايمايدات تحت ظروف مماثلة لظروف تحضير النوفولاك.

تمت معالجة الراتنجات الجديدة باتباع طريقتين تضمنت الأولى إجراء أسترة لمجاميع الهيدروكسيل الفينولية وذلك بمعاملة الراتنجات المحضرة مع كلوريدات حوامض مختلفة بوجود ثلاثي اثيل امين مما اسفر عن تحضير ستة عشر راتنج جديد بينما تضمنت الطريقة الثانية اجراء بلمرة متسلسلة وفق الجذور الحرة للاواصر الفاينيلية الموجودة في حلقات الستراكون ايمايدات المتدلية مما اسفر عن تكوين اربعة بوليمرات متشابكة ثابتة حراريا.

امتازت الراتنجات المحضرة والراتنجات بعد المعالجة بمواصفات جديدة مما يسمح باستخدامها في مجالات مختلفة.