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AMIDATION OF LINEAR LOW DENSITY POLYETHYLENE TO PREPARE INTERPENETRATING NETWORKS WITH SOME TRADE NETWORKS

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Abstract:

Polyethylene was known as an inert polymer and cannot compatible or reacted with other polymers. In this work, the inert linear low-density polyethylene (LLDPE) was grafted by the reactive group maleic anhydride. The grafted polymer can be reacted with alcohols in order to prepare the poly ester of LLDPE. Added of diamine to polyester produce polyamide network. IPNs from the amide networks and networks of commercial epoxy resin and of phenol-formaldehyde polymer were prepared. The chemical structure of the amide networks and their IPNs were studied by FTIR. The results show that there are no chemical reaction between the different networks, but physical association can be present between the network. The thermal properties of the IPNs were investigated. The results show enhancement of the thermal stability of the networks through their IPNs. XRD analysis of the prepared networks many crystalline regions. Many crystalline regions were disappeared or changed their volume and new crystalline regions have been appeared through the preparation of IPNs with phenol formaldehyde or epoxy networks.

Keywords: grafted polyethylene, interpenetrating network, epoxy, phenol – formaldehyde.

اميدة البولي إيثيلين الخطي منخفض الكثافة لتحضير شبكات متداخلة مع بعض الشبكات التجارية

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الملخص: يعرف البولي إيثيلين بأنه بوليمر خامل ولا يمكنه التوافق أو التفاعل مع البوليمرات الأخرى. في هذا العمل، تم) بواسطة مجموعة أنهيدريد المالئيك التفاعلية. يمكن LLDPEتطعيم البولي إيثيلين منخفض الكثافة الخطي الخامل (. يضاف ثنائي الامين إلى البولي استر لإنتاج LLDPEتفاعل البوليمر المطعم مع الكحولات من أجل تحضير بولي إستر من شبكات الأمايد وشبكات راتنجات الإيبوكسي التجارية وبوليمر الفينول IPNsشبكة البولي امايد. تم تحضير

. أظهرت النتائج FTIR الخاصة بها بواسطة IPNفورمالدهايد. تمت دراسة التركيب الكيميائي لشبكات الأمايد وشبكات
عدم وجود تفاعل كيميائي بين الشبكات المختلفة، ولكن يمكن أن يكون هناك ارتباط فيزيائي بين الشبكات. تم دراسة
الخاصة بها. IPN. أظهرت النتائج تعزيز الاستقرار الحراري للشبكات من خلال شبكات IPNsالخواص الحرارية لـ
للشبكات المحضرة بالعديد من المناطق البلورية. اختفت العديد من المناطق البلورية أو تغير حجمها وظهرت XRDتحليل
باستخدام شبكات الفينول فورمالدهايد أو الإيبوكسي.IPNsمناطق بلورية جديدة من خلال تحضير

الكلمات المفتاحية: البولي إيثيلين المطعم، الشبكة المتداخلة، الإيبوكسي، الفينول – الفورمالديهايد.

Introduction:

Polyethylenes (PEs) are currently the most applicable synthetic polymers in the world. (1,2,3). According to their inert and stable hydrocarbon backbone, they can resist the environment conditions for many years, thus substantially contributing to environmental pollution (4,5, 6). Consequently the developments for chemically and thermally resistance has received more intentions from both academia and industry. (7, 8, 9, 10). Grafting of the inert polymer with an active moiety is a technique used to improve the rheological properties of the polymers (11). The grafted polymers have many applications, as for blending with other polymers or forming interpenetrating polymers (12,13). Using of maliec anhydride as an active moiety or crosslinking agent for polyethylene has great interest at recent days, whereby it can it can provide effective groups to improve the morphology, chemical and physical properties of the polymer (14,15).

The interpenetrating polymeric networks (IPNs) system are a combination of more than one polymeric networks in one phase (16). (IPNs) system based on polyethylene and many crosslinking species have been investigated by many researchers (17). IPN of polyethylene- polystyrene was synthesized by using in situ method. The electron microscopic results show a low crystallinity of polyethylene in the product(18). A semi interpenetrating polymer network of polyethylene oxide was used as a source of poly ionic liquid for lithium metal batteries (19). The degradation behavior of polyethylene - polymethacrylate interpenetrating network was studied through the change in the chemical structure of the polymer (20).

In this work, in order to prepare highly thermally stable IPNs from polyethylene, polyethylene was grafted with maleic anhydride and crosslinked with different diamine to get amide networks. IPNs was synthesized from the amide networks with epoxy and phenol – formaldehyde networks.

Experimental:

Materials:

Table (1) shows the chemicals which were used as received

Equipment:

F.T.IR spectra were accomplished by using (BRUKER F.T.IR Infrared. Thermal analyses were measured by using Thermogravimetric Analysis / Q 600 /TA , the heating speed of measuring TGA and DTA of the prepared samples was 20°C \ min. X- ray diffraction was carried out by using XRD- 6000 Shimadzu.

Table (1): The used chemicals

Material	Sources
Linear low density polyethylene (LLDPE)	Labtech.
Maliec anhydride	B.D.H
Dicumyl peroxide	Labtech.
Xylene	Fluka
Toluene	B.D.H
Methanol	B.D.H
Sodium hydroxide	Fluka
Ethylene diamine	B.D.H
1,3 diamino propane	Fluka
Epoxy	Labtech
N,N dimethyl aniline	Fluka
Phthalic anhydride	B.D.H
Phenol	Fluka
Formaldehyde	B.D.H

Methodology:

Grafting of linear low density polyethylene (LLDPE) with maleic anhydride(MAn):

The reaction was carried out in a three necked flask equipped with nitrogen inlet, thermometer, reflux condenser, 10g of (LLDPE) was dissolved in 60 ml of xylene under reflux, certain amount of MAn and (0.1 g) of dicumyl peroxide as an initiator was added and bubbled for 5 min. with nitrogen gas (table 2). The reaction was carried out for 2hrs.The grafted polymeric product was precipitated from methanol, filtered, washed with acetone and dried under vacuum at 50°C. another experiment was followed by adding two portions of the initiator. The second portion (0.1g) was added after a period of 2hrs.

Table 2:grafting of LLDPE with Man

NO.	LLDPE/ g	Man /g	Dicumyl peroxide/g
1	10	10	0.1
2	10	10	0.1/0.1
3	10	20	0.1
4	10	20	0.1/0.1

Evaluation of grafting degree:

The degree of grafting of LLDPE with MAn was determined by titrating the acid groups with alcoholic NaOH.

Preparation the ester from monoalcohol:

1 g of the grafted LLDPE (sample 4) dissolved in 60ml of toluene. Equivalent mole of the methanol to MAn was added in presence of two drops of concentrated H_2SO_4 . The mixture was refluxed for a period of 2hrs., the mixture was left with shaking at room temperature to obtain a suspension solution, which will used in the preparation of polyamides.

Preparation of the polyamide network:

After preparation the ester from mono alcohol, the mixture is concentrated to half under reduced pressure, quantity of amine is added gradually (table 3), mixing the mixture for 24hrs., until yellow gelatinous was appeared, (20 ml) of toluene and reflux the mixture for 15 minutes. The precipitated network was filtered, washed with methanol and dried under reduced pressure.

Table 3: Used diamine for amide networks preparation from sample 4 of grafted LLDPE

No.	diamine	wt./ g
1	Ethylene diamine	0.4
2	1,3 diamino propane	0.45

Preparation of IPNs with phenol-formaldehyde:

Mixed thoroughly 1g of grafted LLDPE (sample 4) with 0.57g phenol and 0.1g of p-toluene sulfonic acid until homogeneity. 0.18ml of 30% formaldehyde solution and equivalent moles of diamine to MAn in the sample 4 of LLDPE were added to the homogenous mixture. Then put the mixture in the microwave oven at 600 watt for different periods (table 4) until complete curing.

Table 4: IPN of amide of LLDPE with phenol formaldehyde network

No.	diamine	(Wt /g)	Time of curing
1	Ethylene diamine	0.73	10 min
2	1,3 diamino propane	0.9	35 min

Preparation of IPNs with Epoxy:

a- Epoxy cured with maleic anhydride grafted the LLDPE:

Mixture of 1g MAn grafted LLDPE (sample 4) with half amount of the needed diamine to complete the network was dissolved in 40ml toluene and refluxed for 1hr. then 0.3g of epoxy resin was added in presence of few drops of N,N-dimethyl aniline as a catalyst, complete the reflux for another 3/4 hrs.. The formed IPN was precipitated from methanol, filtered and dried under vacuum.

b- Epoxy cured with phthalic anhydride:

Mixture 1g of MAn grafted LLDPE (sample 4) and 0.366g of ethylene diamine was dissolved in 30ml toluene was refluxed for 1hr., in another flask a mixture of 0.93g epoxy resin and 0.46g of phthalic anhydride was heated until all the anhydride dissolved, then added to the first mixture of LLDPE in presence of few drops of N,N-dimethyl aniline. The mixture was refluxed for 1hr. The product was precipitated from methanol, filtered and dried in oven at 150°C for 0.5hr. to complete the curing.

Results and Discussion

FTIR,TGA,DTA and XRD were measured for the grafted LLDPE with MAn and all their networks and IPNs

FTIR analysis:

Table 5 shows the signs of the prepared amide networks from LLDPE

Table 5: Signs of the prepared recipes

Sign	recipes
L	Linear low density Polyethylene Pure
LM	Maleic anhydride grafted Linear low density Polyethylene
M1	Network polyester with (Ethylene Diamine)
M2	Network polyester with (1,3 diamino propane)

Figure 1 is the IR spectrum of LLDPE. It show the bands appeared at 2914cm^{-1} and 2847 cm^{-1} which are related to the stretching vibration of aliphatic CH group, while the vibrations appeared at 2914cm^{-1} and 2847 cm^{-1} are belong the bending vibrations of aliphatic CH group. The peak at 730 cm^{-1} is belong To C-C vibration.

The results of grafting of LLDPE with MAn shows that the higher grafting percent was obtained when the initiator was added twice as shown in table 6.

We have choose sample (4) to complete the study where it has higher percent of grafting.

Table 6: Grafting of LLDPE with Man

NO.	LLDPE/ g	Man /g	Dicumyl peroxide/g	Reacted MAn/g
1	10	10	0.1	6.95
2	10	10	0.1/0.1	8.11
3	10	20	0.1	11.76
4	10	20	0.1/0.1	15.37

Figure (2) represent the IR spectrum of sample 4. The absorbance frequencies at 1713cm^{-1} and 1633 cm^{-1} which are belong to the stretching vibration of carbonyl group prove the success of grafting.

Figures (3 and 4) represent the IR spectra of the networks LLDPE with the different uses diamine. Table 7 indicates the bands of the different groups of the networks.

Table 7: . Assignment of the IR-absorption of different amide networks of LLDPE

Sample	C-H str.	C-H bend.	C=O	N-H	C-C
M1	2915 2848	1462 1435	1639(I) 1562 (II)	3348	719
M2	2916 2848	1462 1431	1628 (I) 1553 (II)	3354	771

Thermal analysis:

The DTA, TGA and DSC of the prepared networks and their IPNs are accomplished at a heating rate of 20°C/ min. The results show that the thermal stability of the networks were enhanced when they are converted to IPNs. The amide network M1 shows thermal stability until 140°C, where it loss 25.41% of its weight. The pyrolysis of weight loss of about 55.14 %, at 235 °C (figure 5). The DTA curve shows that the sample started decomposition completely between 400°C until 540°C.

Where by the DSC curve show the Tg is about 150°C. Table (8) indicates the thermal behavior of M1 at different temperature.

Table (8):Thermal analysis of M1 and its IPNs

Network M1		IPN with epoxy cured with MAn		IPN with phenol formaldehyde		IPN with epoxy cured with phthalic anhydried	
T/°C	%	T/°C	%	T/°C	%	T/°C	%
T_{140}	25.41	T_{140}	7.05	T_{100}	7		
$_{235}$ T	55.14	T_{260}	11.88	$_{200}$ T	20		
$_{400}$ T	63.31	$_{420}$ T	48.5	T_{385}	40	T_{415}	49.81
$_{800}$ T	95.74	T_{510}	80.79	T_{500}	78	T_{520}	72.91
		T_{800}	96.83	T_{800}	98.61	T_{800}	96.74

In the other hand, the polyamide of M2 which was crosslinked with 1,3 diamino propane gives other thermal characteristics. TGA and DTA (figure 6) indicate that the sample loss about 10.33% of its weight at 115°C,. and loss 27% at 250°C and started complete decomposition at 375°C. The DSC curve show that the Tg is about 475°C.Table (9) shows the thermal history of M2.

Table 9: Thermal analysis of M2 and its IPNs

Network M2		IPN With Epoxy		IPN with phenol formaldehyde	
T/C	%	T/C	%	T/C	%
T_{115}	10.33	T_{140}	5.1	T_{150}	5
T_{250}	27	T_{252}	20.5	T_{260}	17
T_{360}	37.71	T_{398}	46.76	T_{420}	42
T_{550}	83.64	T_{530}	87.98	T_{525}	79
T_{800}	95.55	T_{800}	97.45	T_{800}	92.26

On the other hand, the thermal analysis for IPN of M1with phenol formaldehyde show thermal stability more than M1. Figure (7) reveals that the polymer loss 7% of its weight at 100°C and start complete decomposition at 540°C. DSC curve show that the Tg is about 506°C while in M1 was 476°C. Table 8 shows the thermal history for this IPN. While the TGA analysis for IPN of M2 with phenol formaldehyde (table 9 and figure 8) shows more thermal stability than M1 with phenol formaldehyde. It loss only 5% of its weight at 150°C and start complete decomposition at 525°C.

Figures 9, 10 and 11 indicated the thermal behaviors of the IPNs of epoxy networks with the amide networks (M2-M1). The results reveals that the IPNs have thermal stability more than the amide networks. Tables 8 and 9 reveals the thermal history of these IPNs. These results emphasize the complete interaction between the two types of networks.

X-ray diffraction analysis:

The X- ray diffraction was carried out for measuring the angle (2θ) at range(10°-90°) and scan speed 3° / min using filter of type (Cu- K beta). The used current is of 30 μÅ and the voltage is 40Kv. The wave length can be calculated by using Bragg law:

n□=□□d.Sinθ

where; n= number of waves , □= wavelength , d= the distance between the surfaces of the crystal lattice, θ = the value of the half-angle at the top of the wave, and the volume of the crystal (V) was calculated according to the equation:

$$V = \frac{0.9\lambda}{B.Cos\theta}$$

Where is B the width of mid-top.

It is well known that the LLDPE have little branches, it's have some degree of crystallinity. Figure (12) illustrate the XRD diagram of M1, its show six region of crystallinity. Grafting with MAn increase the branches on the chains and may decrease the degree of crystallinity. In contrast the crosslinking process can give the opposite result and increase the crystallinity. Table (10) explain the analysis of these regions.

Table (10): XRD analysis of M1 network

NO	Peak 2θ	Interplanner Distdnce dat(ºA)	Height (counts)	B at (deg)	Valueλ	V)Vlaue(
1	11.9568	7.40193	153.37	0.7872	1.54182	1.77238
2	22.2325	3.99862	2293.80	0.4920	1.54178	2.8746
3	23.6258	3.76589	1740.39	0.2952	1.54175	1.3875
4	27.6771	3.22316	785.03	0.5904	1.5418	2.4208
5	32.3132	2.77052	464.95	0.5904	1.5415	2.1114
6	40.7037	2.21487	235.98	0.9600	1.5402	1.5402

It was noticed that the IPN with epoxy network cured with phthalic anhydride (Figure 13 and table 11) that there are two region increase the volume of crystal at 2θ (22 and 24) and disappeared of the crystal regions at(11,23,27,32,40). While the IPN with phenol formaldehyde network show disappearance of crystal at five region but crystal appeared at 21 (figure 14 and table 12).

Table (11): XRD analysis of IPN of M1 with epoxy network (PM)

NO	Peak 2θ	Interplanner Distdnce d at(^{O}A)	Height (counts)	B at (deg)	Valueλ	(V)Value
1	22.2948	3.98760	2988.66	0.3936	1.5416	3.5933
2	24.6810	3.60721	1315.09	0.3936	1.5418	3.6090

Table (12): XRD analysis of IPN of M1 with phenol formaldehyde.

NO	Peak 2θ	Interplanner Distdnce dat(^{O}A)	Height (counts)	B at (deg)	Valueλ	(V)Value
1	21.9099	4.05341	610.23	0.9600	1.5402	1.4708

M2 network have three region of crystal, whereby the long chain bridge can enhanced the folding and aligning the chains. Figure (15) table (13) summarize the results. The IPN with the epoxy network show disappeared of the crystal regions at 2θ (11 and 24) while decrease the degree of crystallinity at 2θ (22) in figure(16) and table (14).

Table (13): XRD analysis of M2

NO	Peak 2θ	Interplanner Distdnce d at(^{O}A)	Height (counts)	B at (deg)	Valueλ	(V)Value
1	11.9159	7.42724	165.22	2.3616	1.54187	2.83587
2	22.2986	3.98692	1499.00	0.4920	1.54187	2.87483
3	24.6242	3.61540	595.22	0.3936	1.54182	3.60868

Table (14): XRD analysis of IPN of M2 with epoxy network

no	Peak 2θ	Interplanner Distdnce d at(^{O}A)	Height (counts)	B at (deg)	Valueλ	(V) value
1	22.1773	4.00513	98.17	0.9600	1.5405	1.4718

Conclusions

1. The grafted LLDPE can be used as a reactive compound to prepare networks .
2. The grafted LLDPE can be reacted with diamine to form network polyamide.
2\. The network polyamide has thermal stability more the grafted LLDPE.
3\. The polyamide can form IPNs smoothly with network of phenol formaldehyde or epoxy .
4\. The prepared IPNs have structural and thermal stability.

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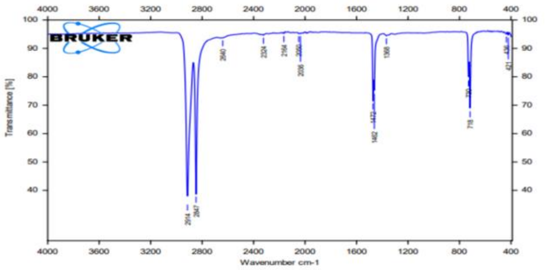	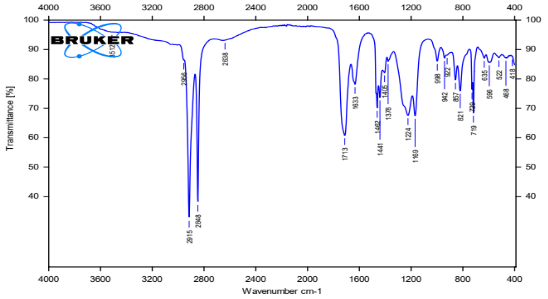
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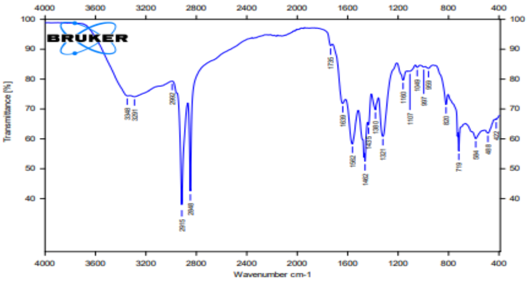	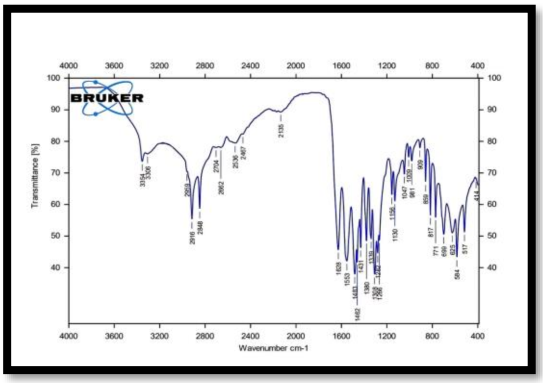
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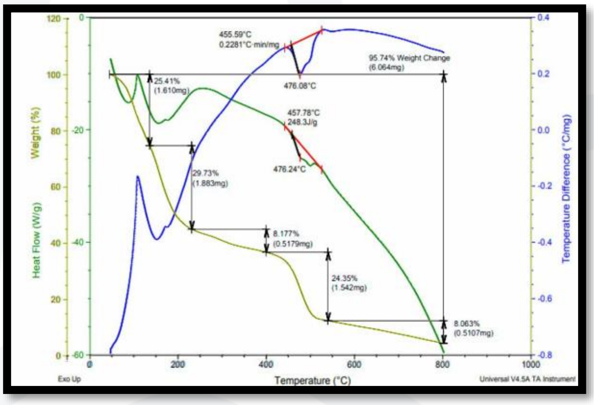	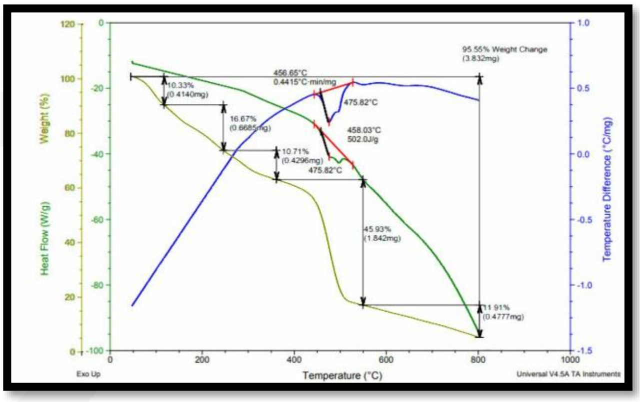
Figure (5): DSC, DTA and TGA Thermogram of M1	Figure (6): DSC, DTA and TGA Thermogram of M2

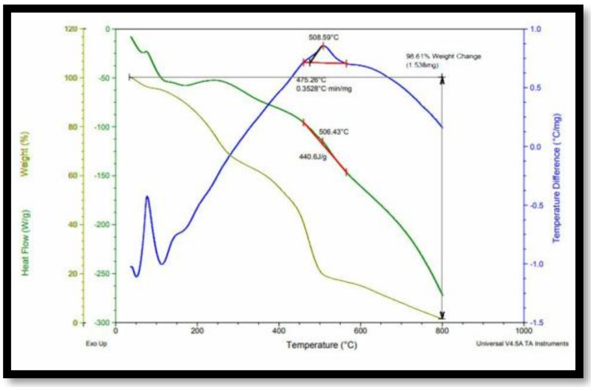	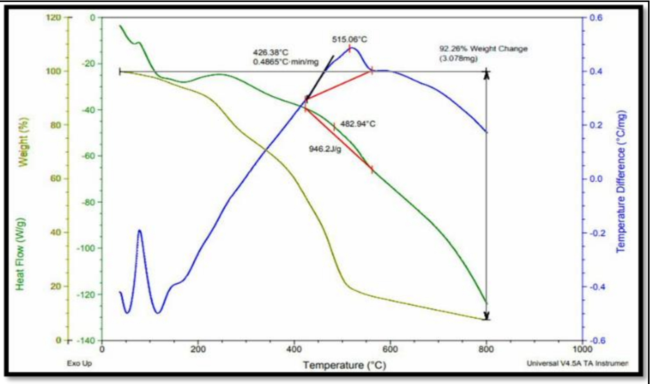
Figure (7): DSC, DTA and TGA Thermogram of IPN of M1 with phenol formaldehyde network.	Figure (8): DSC, DTA and TGA Thermogram of IPN of M2 with phenol formaldehyde network.
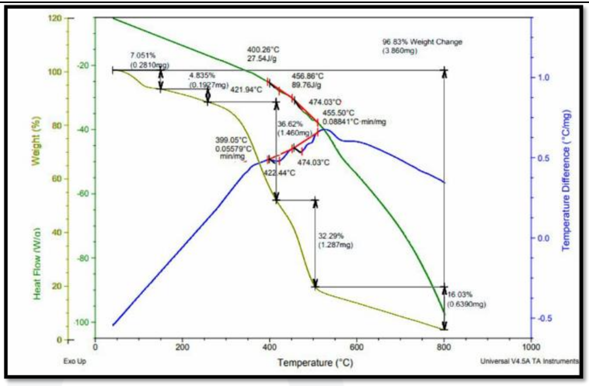	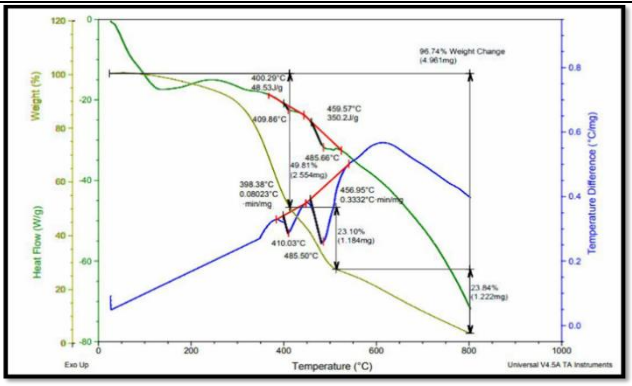
Figure (9): DSC, DTA and TGA Thermogram of IPN of M1 with Epoxy network	Figure (10): DSC, DTA and TGA Thermogram of IPN of M1 with Epoxy network(PM)
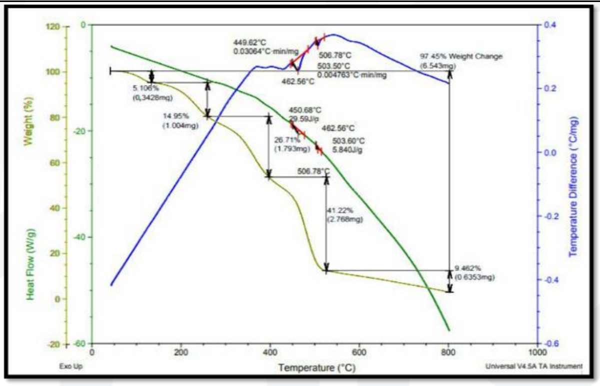	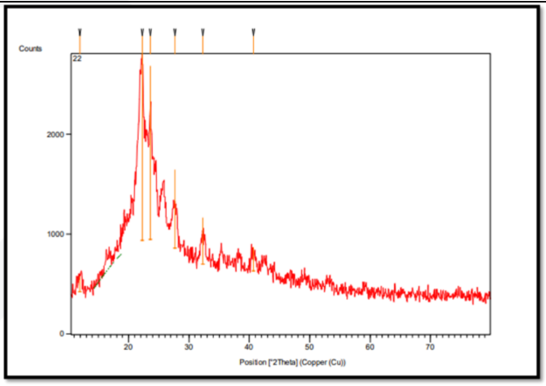
Figure (11): DSC, DTA and TGA Thermogram of IPN of M2 with Epoxy network	Figure (12): XRD diagram of M1.

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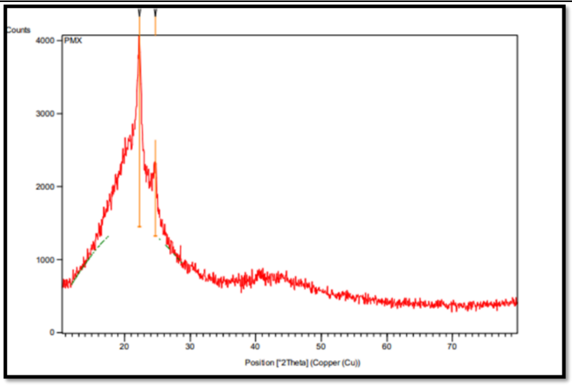
<td>
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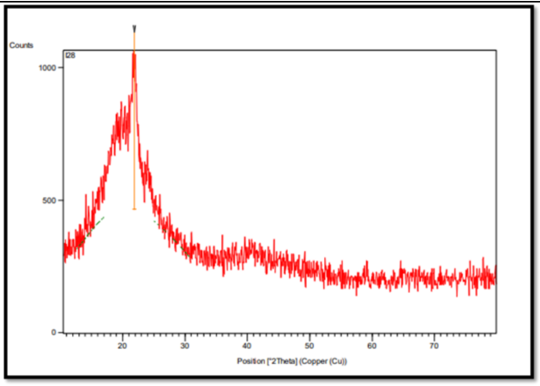
<td>
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<td>Figure (13): XRD of IPN of M1 with epoxy network(PM)</td>
<td>Figure (14): XRD diagram of IPN of M1 with phenol formaldehyde network</td>
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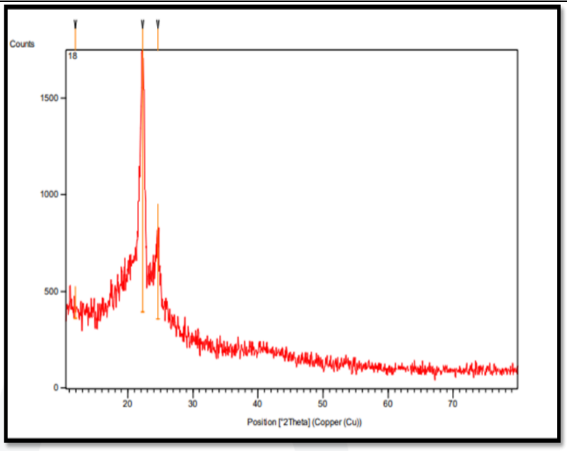
<td>
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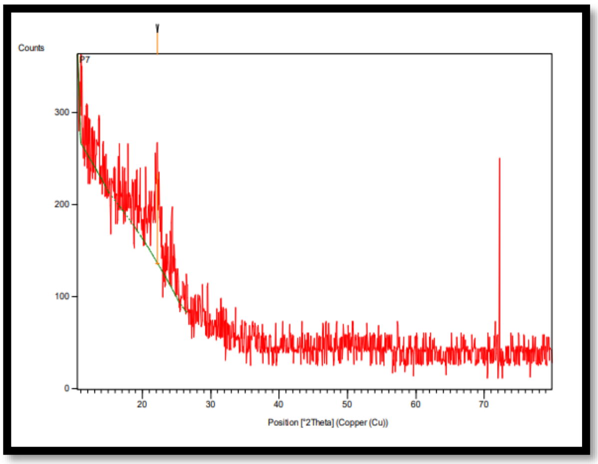
<td>
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<td>Figure (15): XRD diagram of M2</td>
<td>Figure (16): XRD diagram of IPN of M2 with epoxy network</td>
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