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Synthesis and Characterization of N-(4-Nitrophenyl) Maleimide Polymer



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ABSTRACT

This article describes the synthesis and characterization of N-(4-Nitrophenyl) Maleimide polymers. Synthesis monomers were copolymerized with Methyl acrylate (M.A.) and Ethylacrylate (E.A.). Free radical initiators AIBN and BPO were used. The Reactions of the Homopolymer and copolymer were carried out at different times (12,24,30 and 36) hours. The percentage yield was calculated at different solvents and free radical initiator systems. Synthesis Copolymers and Homopolymer and thermal stability were determined by Thermogravimetric analysis. Prepared copolymer and their Copolymer composition were determined by Elemental analysis C, H, N% of monomers, and N% of all the copolymer samples were carried out on Perkin Elmer 2400 by using the combustion's method. With various amounts of Ethylacrylate and Methyl acrylate with N-(4-Nitrophenyl) Maleimide ranging from 0.1 moles to 0.9 moles were performed in a solvent. Structural and molecular characterization of copolymers was performed with FTIR, ¹H-NMR, elemental analysis, and gel permeation chromatography. Number average and weight average Molecular weight were calculated by gel permission chromatography. Free radical polymerization is carried out by the PDI index. Thermal stability was determined by TGA Techniques. The percentage yield was recorded at different time hours.



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INTRODUCTION

N-Substituted Maleimide polymers represent an important class of rigid polymers which have many applications in various industrial fields [1-4]. The presence of imide rings in the backbone of polymaleimides provides these polymers with excellent thermal stability and chemical resistance [5-6]. The polymers of N-Phenylmaleimide and its derivatives have been known to exhibit high T_g due to the rigid imide rings in the backbones. The polymerization of vinyl isocyanate with maleic anhydride is known to be alternating [7] and a similar tendency for the maleimide systems is also observed [8]. Therefore, a well-defined copolymer with a 1:1 composition is formed from the polymerization of a 1:1 mixture of monomers and there is one reactive isocyanates group in every repeating unit of the copolymer. In a subsequent process, the polymers can be functionalized by reaction with the hydroxy group of 4-hydroxy TEMPO. As it will be shown below, these polymers behave as polymeric counterradicals for propagating polymeric radicals. Aromatic polyimides are one of the most important classes of high-performance polymers. Due to their thermal, electrical, and high-temperature mechanical properties, aromatic polyimides have found many applications as high-temperature insulators, coatings, adhesives, and matrices for high-performance composites [9-14]. In the present study, we are focused on the thermal stability of newly prepared homopolymers and copolymers of PNPMI with methyl acrylate, Ethylacrylate.

MATERIALS

THF was purified by distillation after being refluxed for 2 h in the presence of sodium. Dioxane and DMSO were used after distillation. DMF and methanol used in the present work were of analytical grade and were used as received. Maleic anhydride was recrystallized from acetone. Acrylonitrile is used after distillation. Methylmethacrylate (CDH) was shaken two to three times with 5% NaOH to eliminate hydroquinone inhibitor, dried over anhydrous CaCl₂ for 6 hours, and distilled. The head and tail fractions were discarded. AIBN (2,2-azobis-isobutyronitrile (spectrochem.)) was recrystallized twice from methanol before use. BPO (benzoyl peroxide CDH) was used as received.

MEASUREMENTS

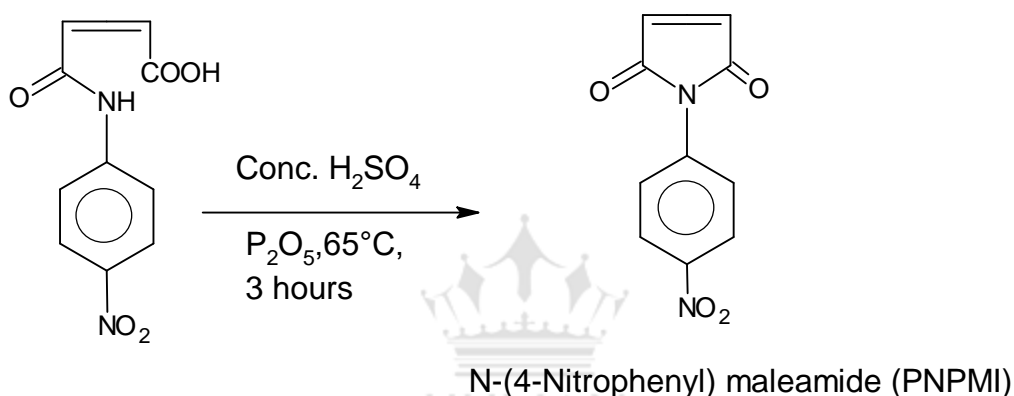
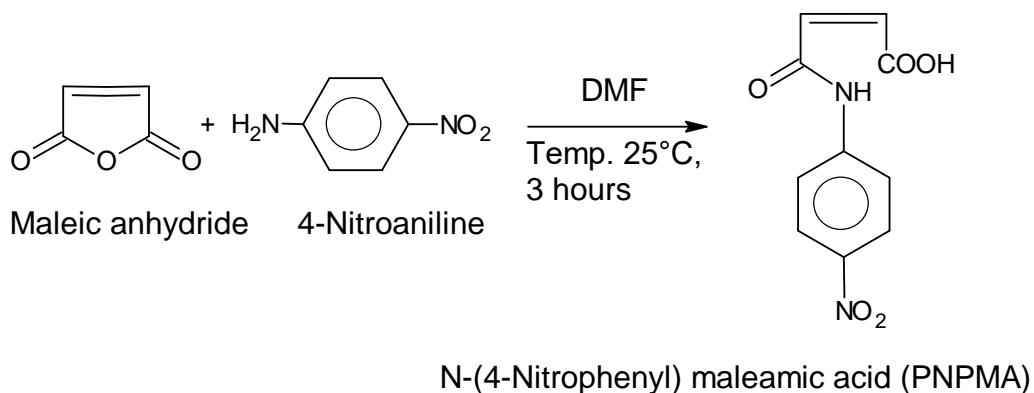
¹H-NMR spectra of monomer and polymer samples were taken in DMSO-d₆ on a Bruker DPX-200/DPX-300 spectrometer at 200/300 MHz. The internal reference used was TMS. FT-IR spectra of the monomer and polymer sample were recorded on a Shimadzu 8201 PC (4000-400 cm⁻¹) FT-IR spectrometer, using the KBr pellet technique. The viscosity measurements were carried out in DMF at 30±0.2 °C, using an Ubbelohde suspended level viscometer. Elemental analysis was made on Carlo Erba Model NA 500 series analyzer. The thermograms in the air were obtained on a Mettler TA-3000 system, at a heating rate of 10 °C/min.

METHODS

Preparation of N-(4-Nitrophenyl) maleamic acid: Nitroaniline (13.8 gm for 0.1mol) and 9.8 gm maleic anhydride were taken in 30ml DMF. The solutions were kept stirring for three hours at 25°C. The reaction mixture was poured into crushed ice. The yellow solid precipitate was obtained, then filtered and dried under vacuum. It was recrystallized from methanol to obtain pure N-(2-Nitrophenyl) maleamic acid. The yield was 70%, m.p. 145°C.

N-(4-Nitrophenyl) Maleimide: Cyclodehydration of the 2-Nitromaleamic acid, an intermediate to maleimide was carried out by treating the former with conc. H₂SO₄ and P₂O₅. The solution was stirred for three hours at 65°C. Then the solution was poured into crushed ice or cooled water, obtained to yellow solid precipitate filtered and washed with water, and dried. N-(2-Nitrophenyl) maleimide was recrystallized from ethyl alcohol solvent. The yield was 65%, m.p. 120°C.

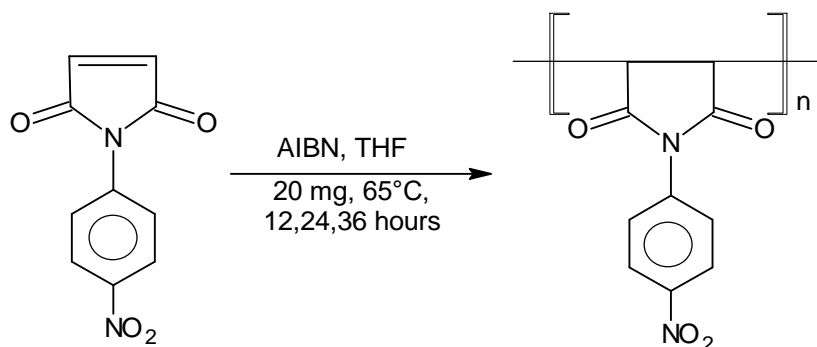
SCHEME -1



Homopolymerization of N-(4-Nitrophenyl) maleimide (PPNPMI)

Polymerization of 4-Nitrophenylmaleimide was carried out in THF solvent using radicals initiator AIBN 65°C. 4-Nitrophenylmaleimide (2.18 gm, 0.01 mol) and THF in 30 ml were placed in a round bottom flask with a reflux condenser. To this solution 20 mg AIBN was added and the reaction mixture was refluxed on heating metal at 65°C for 12, 24, 36, and 48 hours. No polymer or very less polymer was formed if the reaction was carried out for up to 12 hours. The yield was obtained at 32.33% for 24 hours. The synthesized polymer was isolated in the excess quantity of methanol-water mixture. It was dried under vacuum at 55°C.

SCHEME - 2

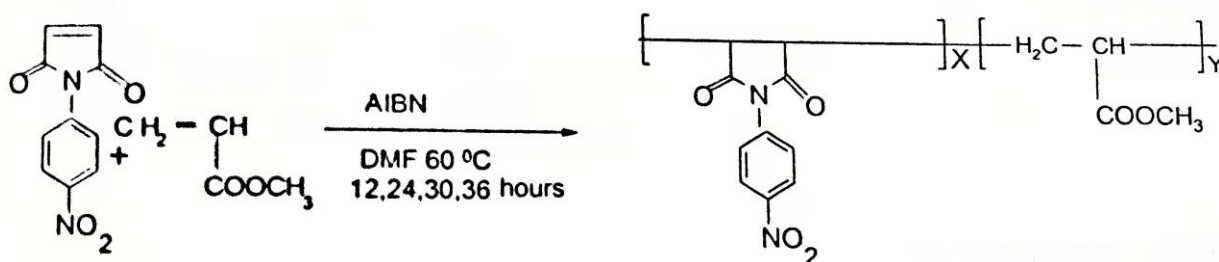


Copolymerization of N-(4-Nitrophenyl) maleimide with MA

Calculated equal amounts of 4-Nitrophenylmaleimide and MA in 30 ml THF solvent were taken in a round bottom flask. The copolymerization reaction was carried out at 65°C for 24 hours. Copolymer samples were isolated in water containing 20 % methanol. The copolymer was purified by first dissolving in THF and then, reprecipitating in the excess quantity of methanol-water mixture. The precipitated copolymers were washed with methanol several times and dried at 60°C under a vacuum. The yield was 40.0%.

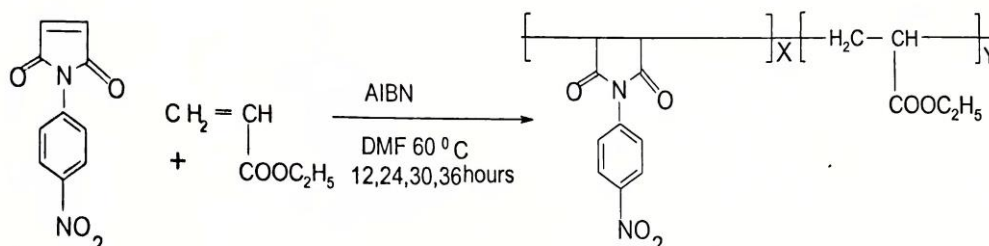
Reaction with Methyl acrylate(PNPMCMA)

SCHEME - 3



SCHEME-4

Reaction with Ethyl acrylate(PNPMCEA)



RESULT AND DISCUSSION

PNPMI

FT-IR spectra (Fig.1, in cm⁻¹) with characteristic absorption frequencies 3091.3,2980.8 (aromatic and alkene C-H stretch), 1797 and 1708 (symmetric and asymmetric stretching of C=O in a five-member imide ring.[15,16] 1634(CH=CH, alkene), 1564 1456 (C=C, aromatic)[17,18].1335,1504 (symmetric and asymmetric stretch of -NO₂) [19]. 967 (CH=CH) [20]. 856(1,4 disubstituted benzene) these are consistent with the structure of PNPMI. ¹H-NMR spectra (Fig.2, 400 MHz TMS, DMSO-d₆, δ, ppm) of PNPMI shows two methines (CH=CH) protons at δ 6.82 (s) ppm; two aromatic protons (o-group in phenyl ring) at δ7.13-7.15 (d) ppm; two aromatic protons (m-group in phenyl ring) at δ7.63-7.64 (d) ppm.

PPNPMI

In Fig. 3, for PPNPMI, the specific characteristic absorption bands observed are at 3088 (C-H aromatic), 2981 (C-H stretch alkene), 1778, 1715 (symmetric and asymmetric stretching of C=O group) 1614, 1651, 1563, 1444, 1596 (C=C stretch aromatic), 1504, 1331 (symmetric and asymmetric, Ar-NO₂ group), 1255 (asymmetric C-O-C stretch), 1110, 1168 (symmetric C-O-C stretch), 856 (1,4 disubstituted benzene) and 690, 605 (out of plane aromatic ring C=C bend) are consistent with the structure of PPNPMI. In Fig.4, the ¹H-NMR spectrum of PPNPMI is observed at δ 7.50–8.08 (d). 2H in the-CH- group appeared at 3.5–4.0 ppm. ¹H-NMR of PPNPMI monomer shows a chemical shift δ at 6.82 (s) ppm characteristic of the CH=CH group.

The disappearance of this in polymer PPNPMI shows that the radical polymerization proceeds through the double bond [21,22,23].

PNPMCMA

The major characteristic absorption bands (in cm^{-1}) are observed at 3190–3050 (Aromatic, C-H stretch, CH_3 , and CH_2 in MMA), 1750, 1790 (C=O symmetric and asymmetric stretch in a five-member imide ring, and C=O stretch of ester), 1602, 1535 (C=C stretch, aromatic), 1487, 1444 (C-H band, $-\text{CH}_2$), 1380 (due to overlapping of C-N stretch and C-H band of $-\text{CH}_3$ group), 1189, 1150 (asymmetric and symmetric C-O-C stretch, 1528 due to CH-stretching of aromatic ring 831 (1,2-disubstituted benzene) and 781, 741 cm^{-1} (out of plane C-H band of a benzene ring, and 699 (out of a plane (aromatic ring C=C band). These characteristic bands confirm that units of both the monomers: PNPMC and MA are present in the copolymer samples. The absence of a characteristic band of the vinyl group at 957 cm^{-1} indicates the polymer formation via the vinyl group.

$^1\text{H-NMR}$ of PNPMCMA data showing In Fig. 5, δ values from 7.5 to 8.1 due to the presence of the phenyl ring and a signal of showing δ at 4.0 it means the presence of MA IN the unit, signal δ values obtained in 3.3 to 3.5 appearance of polymerization proceed via a single bond.

PNPMCEA

In Fig.6, the major characteristic absorption bands (in cm^{-1}) are observed at 3072–2938 (Aromatic, C-H stretch, CH_3 , and CH_2 in MMA), 1712, 1775 (C=O symmetric and asymmetric stretch in a five-member imide ring and C=O stretch of ester), 1602, 1535 (C=C stretch, aromatic), 1487, 1444 (C-H band, $-\text{CH}_2$), 1391 (due to overlapping of C-N stretch and C-H band of $-\text{CH}_3$ group), 1578 due to present of NO_2 , 1615 presence of $-\text{N-C=O}$, 1170 (asymmetric and symmetric C-O-C stretch, 831 (1,2-disubstituted benzene) and 774 cm^{-1} (out of plane C-H band of the benzene ring, and 666 (out of a plane (aromatic ring C=C band). These characteristic bands confirm that units of both the monomers: PNPMI and EA are present in the copolymer samples. The absence of a characteristic band of the vinyl group at 957 cm^{-1} indicates the polymer formation via the vinyl group⁵. In Fig.7, the chemical shift for four aromatic protons

observed δ at 7.5–8.1 ppm and 2H (–CH–CH–), –OCH₃ observed δ at 3.4–3.7 ppm appearance of –CH-CH-proton and –OCH₃ Protons.

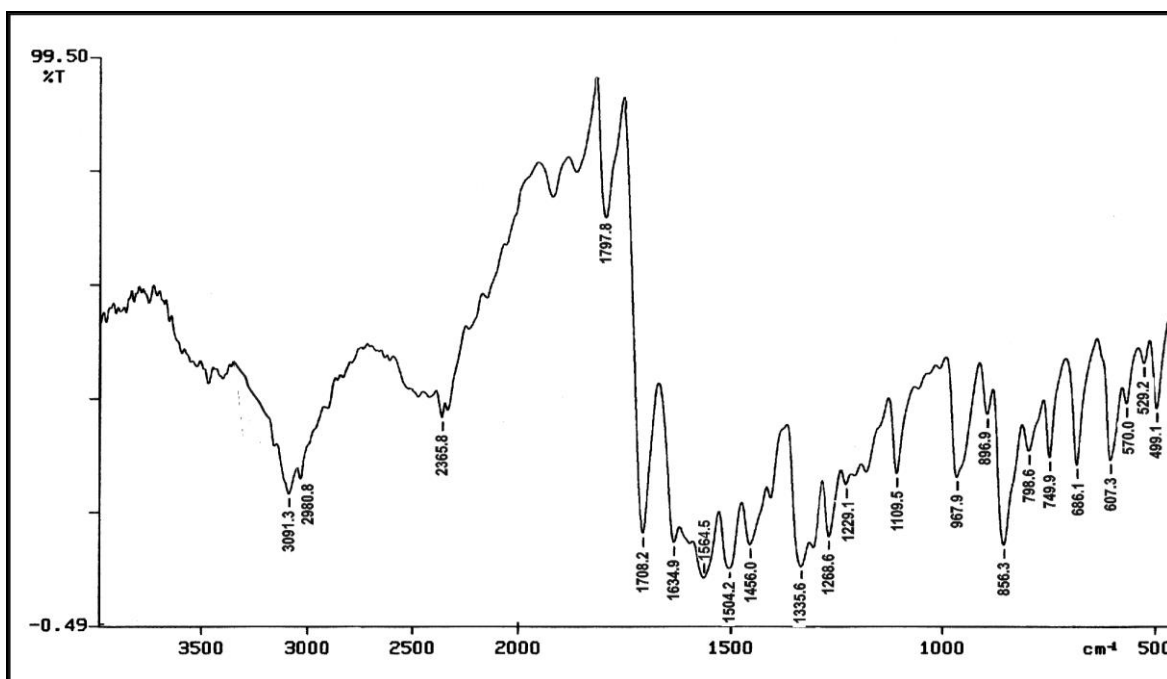


Figure.1. of PNPMI

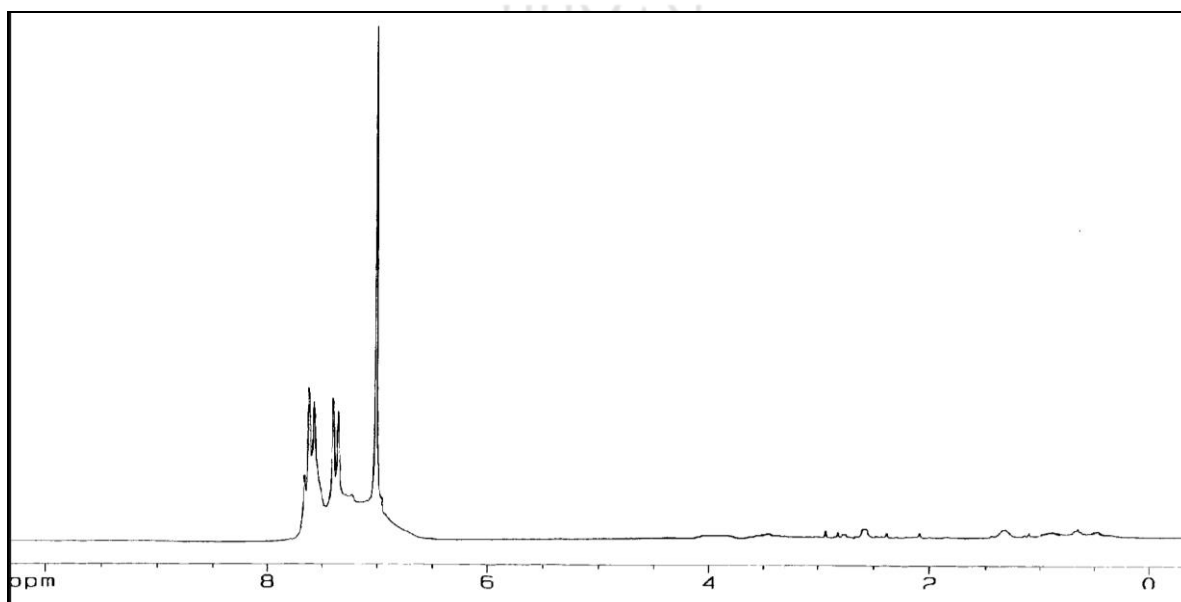


Figure .2. FTIR Spectra of PNPMI

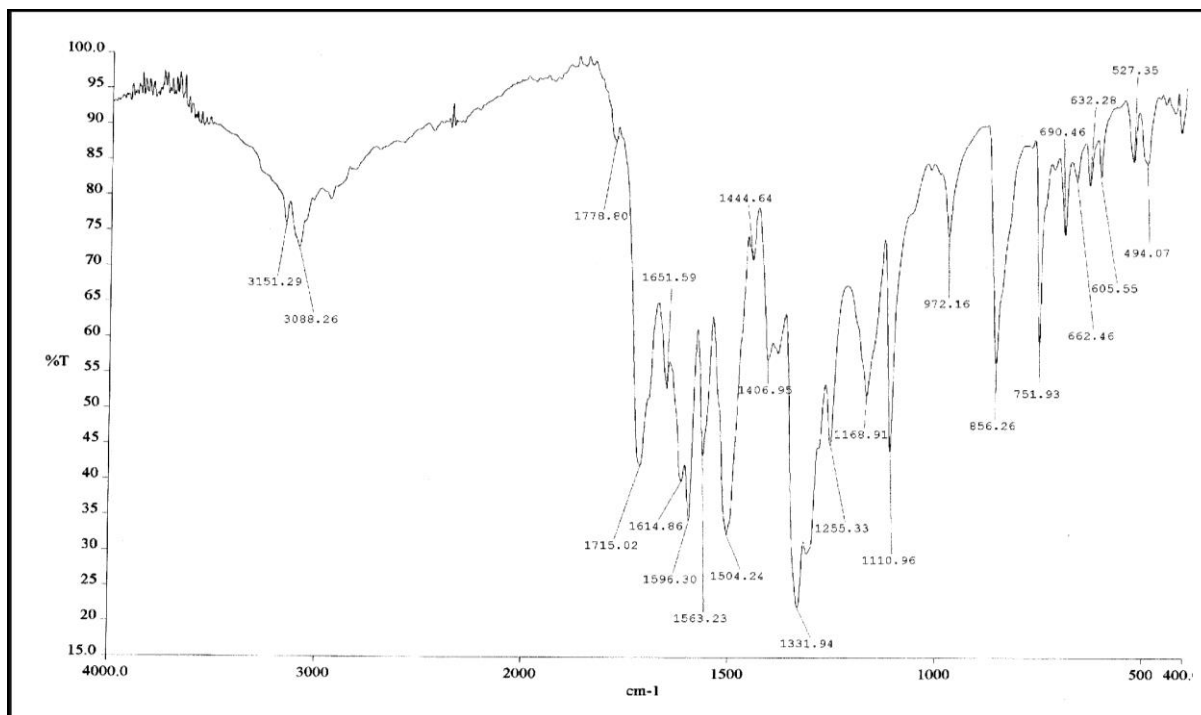


Figure.3. FTIR Spectra of PPNPMI

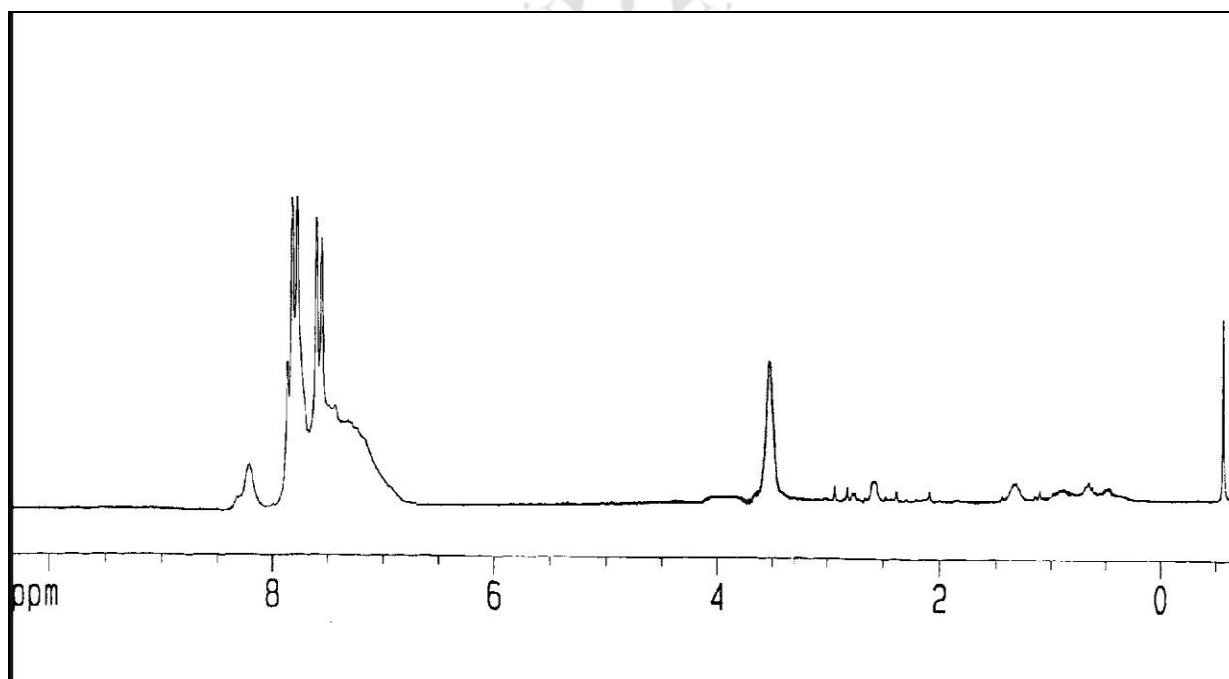


Figure.4. of ¹H NMR Spectra of PPNPMI

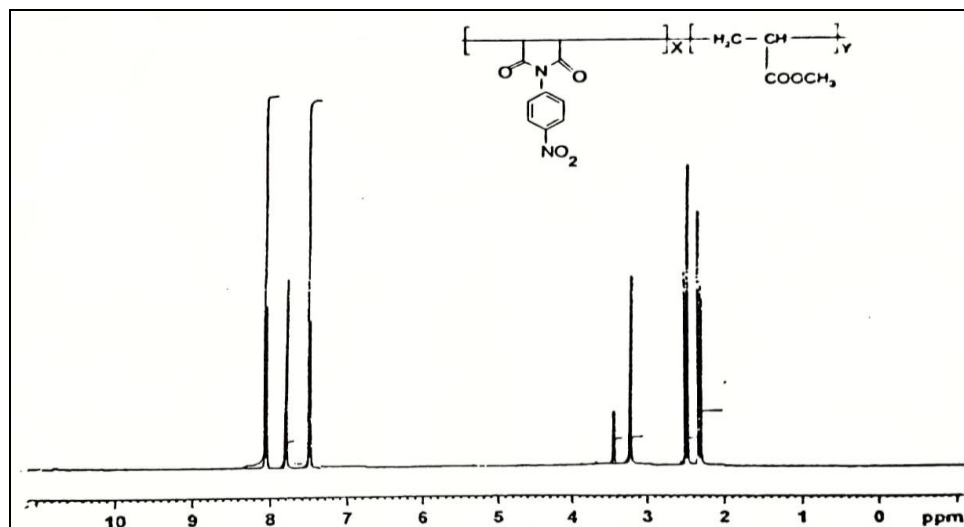


Fig.5. for ¹H-NMR Spectra of PNPMCMA

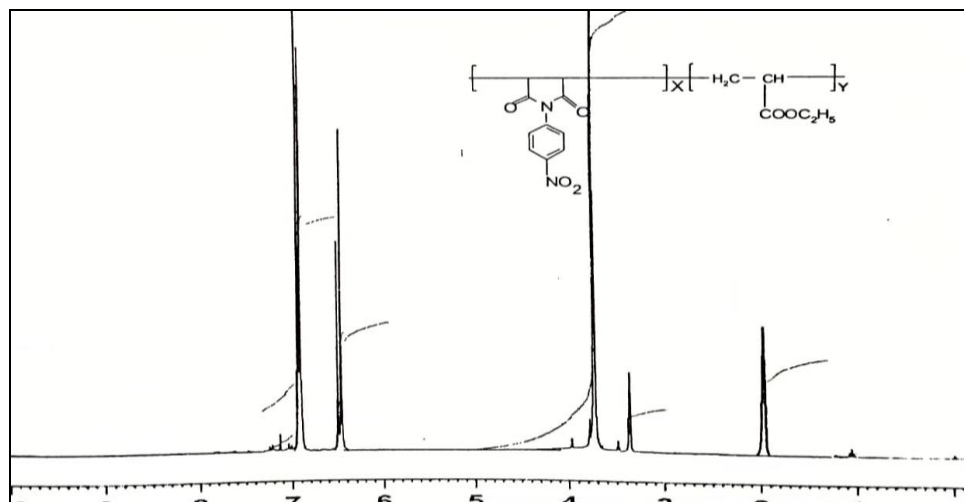


Fig.6. For ¹H-NMR Spectra of PNPMCME

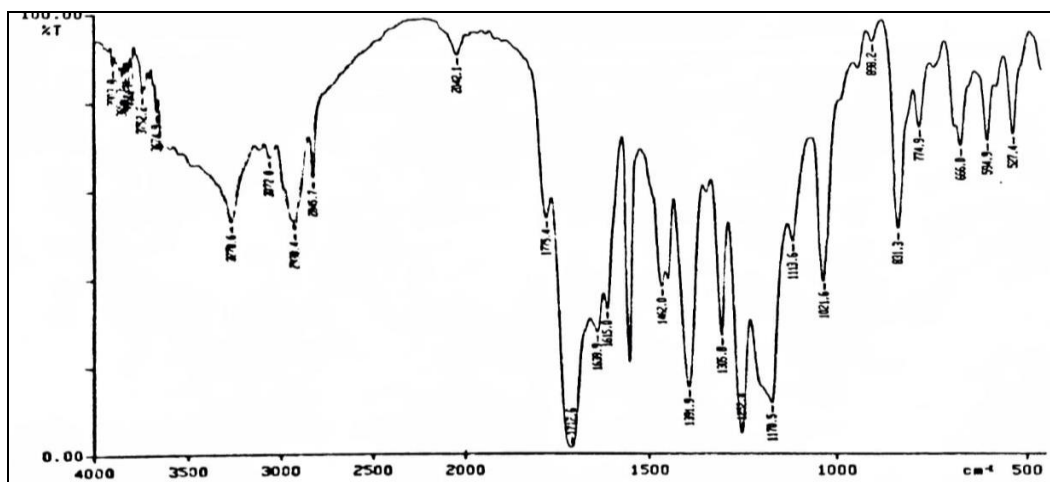


Fig. 7. FT IR Spectra of PNP MCEA

Viscosity behavior in solvent

The increase in viscosity is caused by strong internal friction between the randomly coiled and swollen macromolecules and the surrounding solvent molecules. Solvent viscosity of resins is influenced by the concentration of resin, the softening point, the molecular weight distribution, the chemical composition of the resin, and the type of solvent. The higher the resin concentration, the higher the viscosity. As the size of the monomer increases intrinsic viscosity increases. Intrinsic viscosity evaluation by following relations. The flow time of the concentration was measured by the Ubbelohde viscometer, where the flow time of solution (t) and solvent t_0 ,

The specific viscosity was determined by following relations this was used to find reduced

$$\text{viscosity, } \eta_{\text{reduced}} = \frac{\eta_{\text{specific}}}{C}, \quad \eta_{\text{sp}} = \frac{t - t_0}{t_0}$$

The intrinsic viscosity $[\eta]$ was obtained from the plots of reduced viscosity versus concentration extrapolating the curve at $c \rightarrow 0$. Intrinsic viscosity (η)

Table.1 Typical data of viscosity measurements copolymer of PNPMCMA in DMF at 30°C

Polymer code	Conc.(c) (g/dl)	$\cdot \eta_{rel}$	η_{sp}	$\eta_{sp/c}$ (dl/g)	Intrinsic Viscosity
PNPMCMA	0.1	1.0291	0.0291	0.291	
	0.2	1.0590	0.0590	0.295	
	0.3	1.0915	0.0915	0.305	0.284
	0.4	1.1252	0.1252	0.313	
	0.5	1.1560	0.1560	0.317	
	0.6	1.1938	0.1938	0.323	

Table.2 Typical data of viscosity measurements copolymer of PNP MCEA in DMF at 30°C

Polymer code	Conc.(c) (g/dl)	$\cdot \eta_{rel}$	η_{sp}	$\eta_{sp/c}$ (dl/g)	Intrinsic Viscosity
PNPMCEA	0.1	1.0302	0.0302	0.302	
	0.2	1.0624	0.0624	0.312	
	0.3	1.0948	0.0948	0.316	0.295
	0.4	1.1304	0.1304	0.326	
	0.5	1.1660	0.1660	0.332	
	0.6	1.2052	0.2052	0.342	

Table. 3 Typical data of viscosity measurements Homopolymer of PMNPMI in DMF at 30°C

Polymer code	Conc.(c) (g/dl)	$\cdot \eta_{rel}$	η_{sp}	$\eta_{sp/c}$ (dl/g)	Intrinsic Viscosity
PPNPMI	0.1	1.0071	0.0071	0.071	
	0.2	1.0177	0.0177	0.0885	
	0.3	1.02805	0.02805	0.0935	0.068
	0.4	1.0388	0.0388	0.0970	
	0.5	1.0533	0.0533	0.1067	
	0.6	1.06786	0.06796	0.1131	

From the comparison of tables 1 to 3, we found that the intrinsic viscosity of Homopolymers is less than that of copolymers it is due to the presence of both units in the copolymers, while homopolymers have only a single unit in their backbone.

Relation of the molecular weight and Viscosity.

Molecular weight was calculated by gel permission chromatography and here it is seen that the molecular weight of the copolymer is more than that of the homopolymer it indicates that copolymers having both are units and Homopolymer have only single repeat units in their backbones.

Typical data of number average and weight average and log values of Homopolymer and copolymer are given in Table 4.

Polymer code	Mw	Log Mw	$\eta \times 10^3$ dl/gm	Log $\eta \times 10^3$	Intrinsic viscosity
PPNPMI	2779	3.4439	42	1.6232	0.068
PNPMCEA	28458	4.4258	295	2.4698	0.295
PNPMCMA	57391	4.7588	232	2.3655	0.232

Effect of Different initiator and Solvents on yield of Polymers.

For the industry's purpose, this is very important which type of initiator is very useful for production yield. Here polymerization processes were carried out in different solvents using a different free radical initiator. Results were summarized in table 5.

Table 5. Percentage Yield of copolymers in different solvents and free radical initiator

Polymer code	Solvent	AIBN	BPO	Colour
PNPMCMA	DMF	47.0 %	46.0 %	Brown
	THF	44.2 %	44.4 %	Yellow

Table 6. Percentage Yield of copolymers in different solvents and free radical initiator

Polymer code	Solvent	AIBN	BPO	Colour
PNPMCEA	DMF	51.9 %	51.0%	Brown
	THF	48.2 %	48.2 %	Yellow

From above the table 5,6 given the conclusion that the percentage yield of PNPMA, PNPME in AIBN-DMF system was more than that of AIBN –THF solvent system. AIBN-DMF System is most suitable system for obtaining the better yield of above polymers.

Effect of reaction time on yield of Homopolymer and Copolymer

Polymerization reaction carried out at different time hours and obtained results are summarized in table 7. Table indicates that percentage yield was increase with time hours and homopolymer shows fewer yields as compare to copolymers.

Percentage yield of Homopolymerization and Copolymerization are given in table 7.

Polymer code	12hrs.	24hrs.	30hrs.	36hrs.	48hrs.
PPNPMI	18.78	33.23	38.12	40.78	42.33
PNPMCMA	42.7	43.8	45.2	47.0	48.80
PNPMCE	45.9	47.0	49.2	51.9	52.0

Relation between Molecular weight and Polydispersity.

The polydispersity index (PDI), is a measure of the distribution of molecular mass and on behalf of the PDI values we find out the polymerization reactions and it indicated the distribution of individual molecular masses in a batch of polymers. Obtained molecular weight and number Average molecular weight of the synthesis Homopolymer and Copolymers and their PDI values are given n table 8.

Table.8 shows the number average and weight average molecular weight of Polymers and the Polydispersity index.

Polymer Code	Number average	Weight Average	Polydispersity	Start Mw
PPNPMI	456	519	1.138	2789.8
PNPMCMA	626	1892	3.021	28458.5
PNPMCE	1119	2361	2.109	28458.5

Solubility Behavior of Polymers

Solubility of any polymer is very useful for research purposes because, on behalf of solubility, any industries classified the polymers, which polymer is easily soluble or not. Recent developments in polymeric delivery systems for the controlled release of therapeutic agents have demonstrated that these systems not only can improve drug stability both in vitro and in vivo by protecting unstable drugs from harmful conditions in the body but also can increase residence time at the application site and enhance the activity duration of short half-life drugs. Therefore, compounds that otherwise would have to be discarded due to stability and bioavailability problems may be rendered useful through a proper choice of the polymeric delivery system [24]. In this article, we selected some important solvents for knowing information about synthetic polymers. The solubility of Homopolymers and copolymers are given in table 9.

Table.9. shows the solubility of Homopolymer and Copolymer and their appearance.

Solvent	PPNPMI	PNPMCMA	PNPMCME	Color
THF	S	S	S	Yellow
DMF	S	S	S	Yellow
DMSO	S	S	S	Yellow
DIOXANE	S	S	S	Yellow
ACETONE	S	S	S	Yellow
ETHYL ACETATE	S	S	S	Yellow
TOLUENE	S	S	PS	Yellow
CCl ₄	S	PS	PS	Yellow
CH ₂ CL ₂	S	S	S	Yellow
METHANOL	S	S	S	Yellow
BENZENE	S	PS	S	Yellow
XYLENE	PS	PS	PS	Yellow
CHCl ₃	S	S	S	Yellow

Thermal Gravimetric Analysis

Synthesis of homopolymer Copolymers and their thermogravimetric analysis obtained by the TGA Techniques. The thermograms were analyses obtained for information about the percentage weight loss at different temperatures. The results of homopolymers and copolymers are summarized.

Table.10. Shows the percentage thermal degradation of homopolymers and copolymers.

Polymer code	Degradation step	T _i °C	T _{max} °C	T _f °C	Residue at 500°C
PPNPMI	I	240	340	370	20.88
	II	370	410	550	
PNPMCMA	I	213	360	448	27.18
	II	448	500	560	
PNPMCEA	I	211	325	400	4.85
	II	400	455	570	

Table.11. Percentage weight loss of homopolymer and copolymer are summarized.

Polymer code	200°C	300°C	400°C	500°C	550°C
PPNPMI	-0.8	-20.8	-72.8	-79.1	-79.2
PNPMCMA	-2.98	-52.29	-78.99	-79.67	-81.28
PNPMCEA	-1.01	-12.45	-81.67	-95.15	-96.24

From the comparison, the above table10 and 11 reveals that the percentage degradation of the copolymer is more as compared to Homopolymer it is due to Homopolymer having only a single repeat unit while the copolymer having both units hence degradation rate in the copolymer is and thermograms are shown in the figure from 8 to 10.

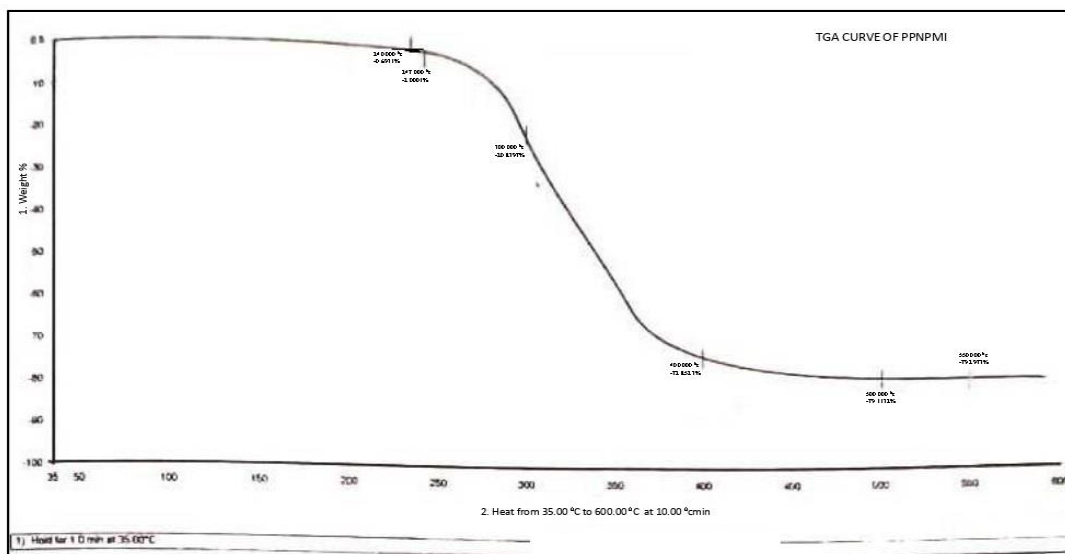


Fig.8. TGA Curve of PPNPMI

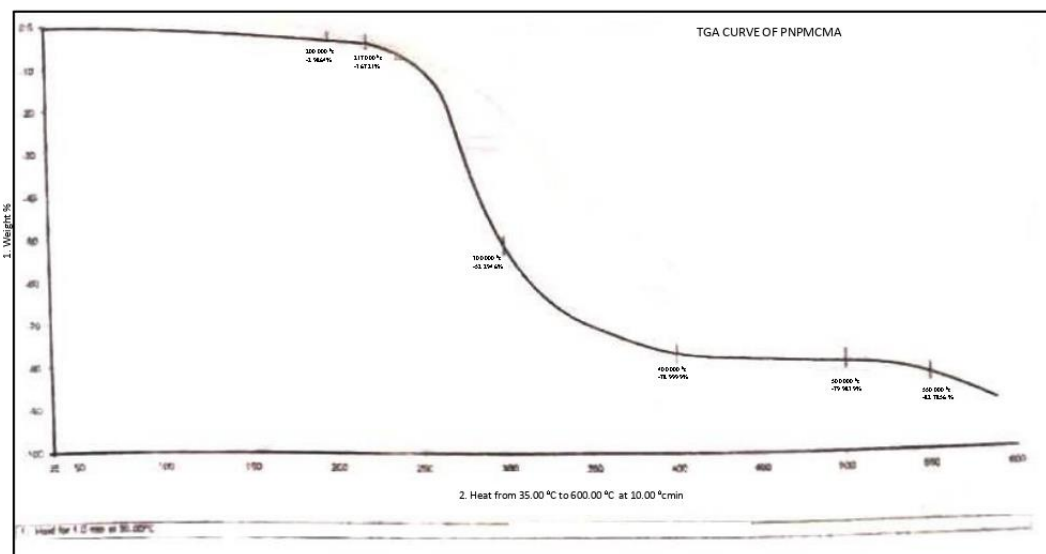


Fig.9 TGA Curve of PNPNCMA

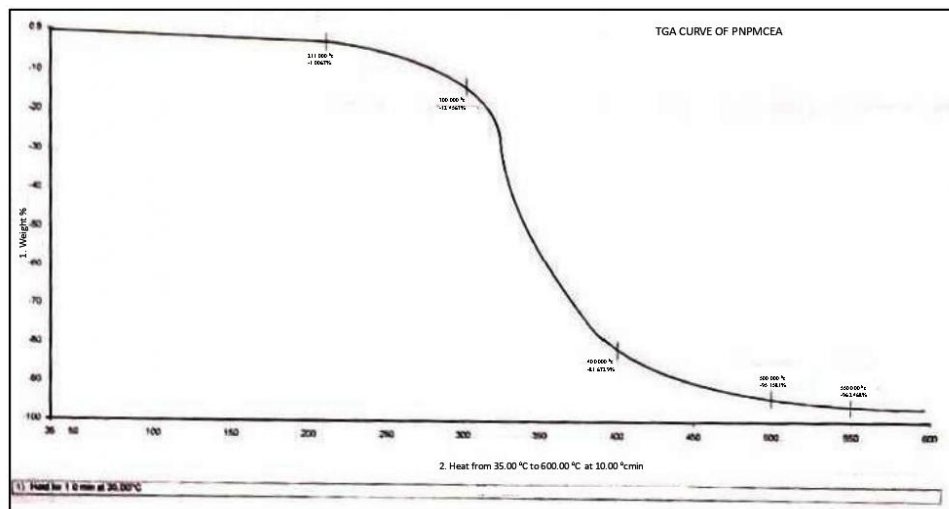


Fig.10 TGA Curve of PNPMECE

CONCLUSION

Synthesis homopolymer and copolymer show excellent solubility in THF, DMF, DMSO, and Chloroform. Ethylacetate. Although some factors which are effected solubility .some factor such as solvent polymer nature, temperature, and molecular weight. texture and length of the polymer. In this article, we conclude that the polymerization reaction proceeds via free radical polymerization. The thermal stability behavior of homopolymer is more stable than copolymer because of this, only a single unit is present in the homopolymer. The molecular weight of the copolymer is more than that of the homopolymer it is due to the copolymer having both units. Weight average and number average molecular weight determined by the Gel permission chromatography.

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