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Synthesis and Characterization of 4-Nitropheny Maleimide and Copolymerized with Acrylonitrile and Acrylamide

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Abstract: ***In this article we are prepared heat resistant polymer containing N-(4-Nitro phenyl) maleimide (PNPMI) and Acrylonitile copolymerization under the identical conditions in DMF and THF solvent. The characteristics of the copolymer were analyzed by Fourier transform infrared spectroscopy, nuclear magnetic resonance spectroscopy, gelpermeation chromatography, and elemental analysis. The thermal stability and degradation mechanism of the copolymer were investigated by Thermogravemetric analysis. Free radical initiator using as an AIBN and BPO in Polymerization reaction , Percentage yield and different solvent system applied and calculated percentage yield. Percentage nitrogen, carbon, and hydrogen content calculate in polymers. The radical polymerization find out by the Polydispersity index. Shape of the polymer in given solution were determined by using range in between according to PDI index. Polymerization process observed by the Polydispersity index. Intrinsic viscosity determined by the using Ubbehold viscometer .Excellent composition structure and Solubility of the Homopolymer and copolymer were determined in different solvent.***

Keywords: Polymerization. TGA, AIBN, BPO, DMF, THF

I. INTRODUCTION

High-performance polymer matrix composites, particularly for aerospace applications, are designed to withstand high thermal and aerodynamic stresses. Among the diverse thermoset resins, bismaleimide resin and Phenylmaleimide polymer stands out as a class of high-performance addition-curable polyimide resin having excellent thermal, physical, and thermo mechanical properties. Thermal behavior of acrylonitrile copolymers is highly depend on the method of polymerization, chemical nature of comonomer and its content. Properties like glass transition temperature , melting and crystallization is affected by the change in crystallinity and perfection. Gupta and coworkers [1] investigated the effect of 2-hydroxy ethyl methacrylate, methacrylonitrile and methylacrylate on crystallinity and crystalline perfection using X-ray and dielectric measurements. The determination of the reactivity ratio has been the major focus of studies described in the open literature for this copolymerization system .[2-11] .In continuation our previous published paper of 2-Nitro phenylmaleimide,3-Nitro Phenylmaleimide and copolymers with various amounts of methylacrylate and methylmathacrylate in copolymer and their comparative study investigated [12]. 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 [13] In present article we are synthesis of newly maleimide monomer of 4-Nitrophenylmaleimide and copolymerized with Acrylonitrile and characterized by the ^{1}H-NMR,FTIR and Thermal analysis by the Thermogravemetric analysis.

II. EXPERIMENTAL

A. Materials

Maleicanhydride were recrystallized from acetone. Methylmethacrylate (CDH) was shaken two to three times with 5% NaOH to eliminate hydroquinone inhibitor, dried over anhydrous CaCl2 for 6 hours and distilled [14]. The head and tail fractions were discarded. AIBN (2,2_-azobis–isobutironitrile (spectorchem.)) was recrystallized twice from methanol prior to use. BPO (benzoyl peroxide CDH) was used as received. 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.

Measurements

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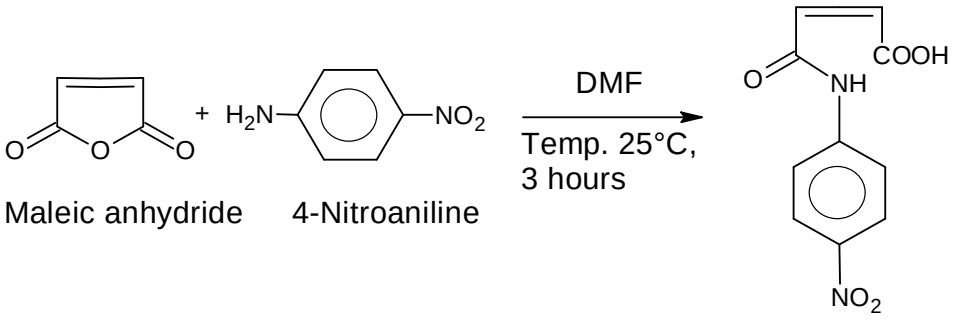
[1]H-NMR spectra of monomer and polymer samples were taken in DMSO-d6 on a Bruker DPX-200/DPX-300 spectrometer at 200/300 MHz. The internal reference used was TMS. FT-IR spectra of the monomer and polymer sample were recorded on a Shimadzu 8201 PC (4000-400 cm-1) FT-IR spectrometer, using KBr pellet 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 air were obtained on a Mettler TA-3000 system, at a heating rate of 10 °C/min

B. Methods

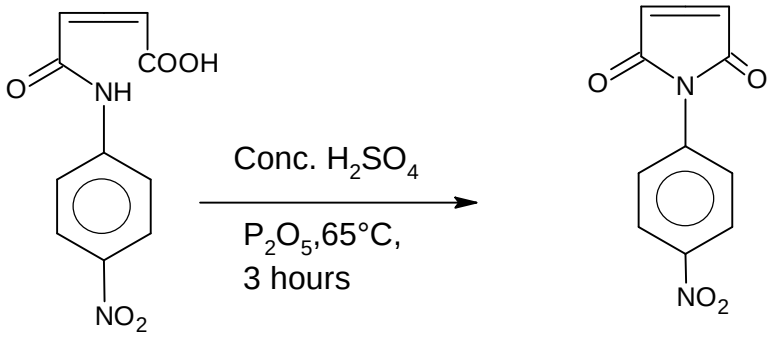
Preparation of N-(4-Nitorphenyl) maieimide (PNPMI): Nitroaniline (13.8 gm for 0.1mol) and 9.8 gm maleic anhydride were taken in 30 ml DMF. The reaction mixture was stirred for three hours at 25°C. The reaction mixture was poured in crushed ice. Yellow solid precipitate was obtained, then filtered and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

N-(4-Nitrophenyl) Maleimide: Cyclodehydration of the 4-Nitromaleamic acid, this solution treated with conc. H_2SO_4 and P_2O_5 and stirred for three hours at 65°C. Then the solution poured in crushed ice or cooled water, obtained to yellow solid precipitate filtered and washed with water and dried.N-(4-Nitrophenyl) maleimide was recrystallized from ethyl alcohol solvent. The yield was 65%, m.p. 120°C.

SCHEME-I



N-(4-Nitrophenyl) maleamic acid (PNPMA)



N-(4-Nitrophenyl) maleamide (PNPMI)

C. Homopolymerization of N-(4-Nitrophenyl) maleimide (HPNPMI)

4-Nitrophenylmaleimide (2.18 gm, 0.01 mol) were taken in 30 ml THF solvent in round bottom flask with reflux condenser. Free radical 20 mg AIBN was added and the reaction mixture was refluxed on heating mental at 65°C for 12, 24, 36 and 48 hours. Product was occurred 32.33% for 24 hours. Prepared polymer was separated in excess amount of methanol water mixture. It was dried under vacuum at 55°C then kept under dedicator free from moisture.

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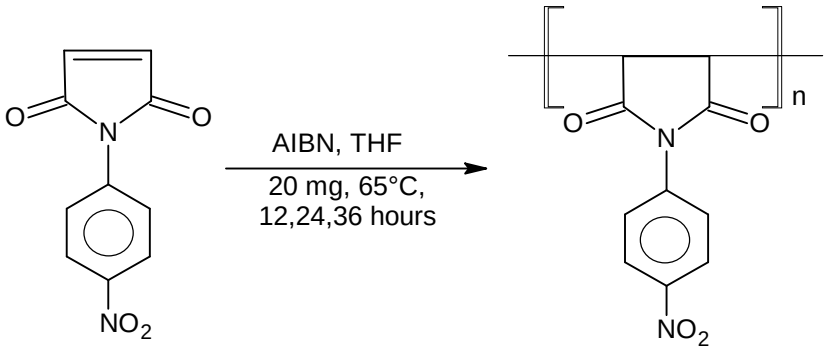
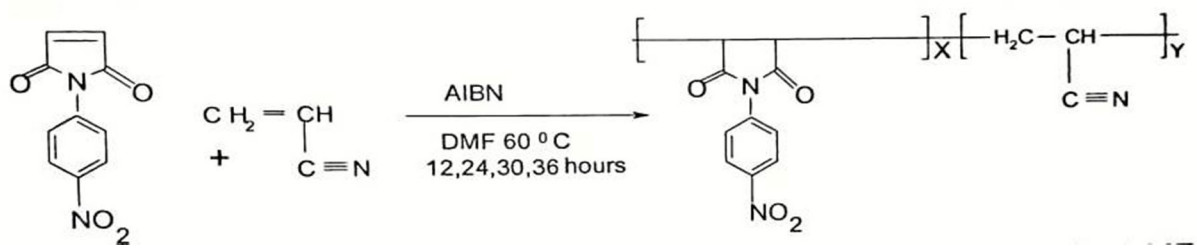
SCHEME - 02



D. Copolymerization of N-(4-Nitrophenyl) maleimide with Acrylonitrile (PNPMCAN)

Equal amount of 4-Nitrophenylmaleimide and Acrylonitrile in 30 ml THF solvent were taken in a round bottom flask. The reaction was carried out at 65°C for 12, 24 ,36 hours. Copolymer samples were isolated in water containing 20 % methanol. The copolymer were purified by first dissolving in THF and then, reprecipitating in excess quantity of methanol water mixture. The precipitated copolymers were washed with methanol several times and dried at 60°C under vacuum.

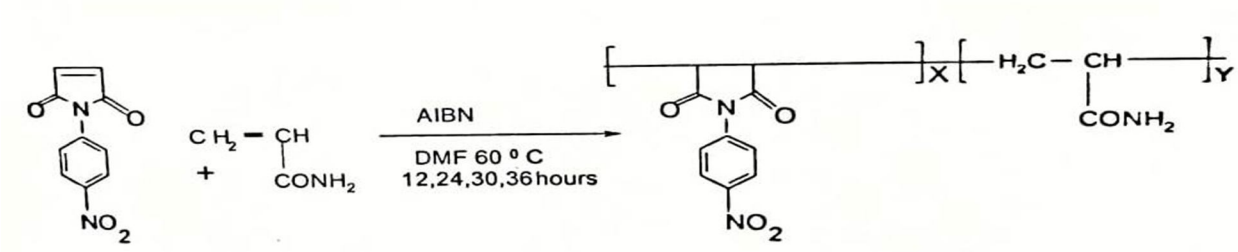
SCHEME-3



E. Copolymerization of N-(4-Nitrophenyl) maleimide with Acrylamide (PNPMCAA)

Equal amount of 4-Nitrophenylmaleimide and Acrylamide in 30 ml THF solvent were taken in a round bottom flask. The reaction was carried out at 65°C for 12, 24 ,36 hours. Copolymer samples were isolated in water containing 20 % methanol. The copolymer were purified by first dissolving in THF and then, reprecipitating in excess quantity of methanol water mixture. The precipitated copolymers were washed with methanol several times and dried at 60°C under vacuum.

SCHEME-4



III. RESULTS AND DISCUSSION

A. PNPMI

FT-IR spectra (Fig.1, in cm^{-1}) 3091.3, 2980.8 (aromatic and alkene C-H stretch), 1797 and 1708 (C=O) [15,16]. 1634(CH=CH), 1564 1456 (C=C, aromatic) [17,18].1335,1504 (symmetric and asymmetric stretch of $–NO_2$) [19]. 967 (CH=CH) [20]. 856(1,4 disubstituted benzene) these are consistent with the structure of PNPMI. ^{1}H-NMR spectra (Fig.2, 400 MH_z TMS, DMSO-d_6, δ, ppm) of PNPMI shows two methine (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 proton (m-group in phenyl ring) at δ7.63-7.64 (d) ppm .

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B. HPNPMI

In Fig. 3, for HPNPMI, 3088 (C-H aromatic), 2981 (C-H stretch alkene), 1778, (C=O group) 1614, 1651, 1563, 1444, 1596 (C=C stretch aromatic), 1504, 1331 (symmetric and asymmetric, Ar-NO_2 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 HPNPMI. In Fig.4, the ^{1}H–NMR spectrum of HPNPMI is observed at δ 7.50–8.08 (d). 2H in –CH–CH– group have appeared at 3.5–4.0 ppm. ^{1}H–NMR of PPNPMI monomer shows chemical shift δ at 6.82 (s) ppm characteristic of CH=CH group. The disappearance of this in polymer PPNPMI shows that the radical polymerization proceed through the double bond [21-23].

C. PNPMCAN

In fig.5, 3937.3 (Aromatic,C-H),3278 (CH=CH), 1718, 1775 (C=O), 1559 (-NO_2 group) 1461.9 (C-H band, –CH_2), 1394,1301 (C-N, Stretching of maleimide), 1174 (C-O-C), 1560 (-NO_2), 832 (1,2-disubstituted benzene) and 774.3 cm^{-1} (out of plane C-H band of benzene ring), and 668 (out of plane (aromatic ring C=C band). ^{1}H-NMR of PNPMCAN, In Fig.6, δ values from 7.5 to 8.1 due to present of phenyl ring and a signal of showing δ at 4.0 present of AN, δ values obtained in 2.8 to 3.6 ppm, polymerization precede via single bond.

D. PNPMCAA

In fig.7, 2931 (Aromatic,C-H),3092 (CH=CH), 1718, 1709 (C=O), 1559 (-NO_2 group) 1484 (C-H band, –CH_2), 1384,1301 (C-N, Stretching of maleimide), 1188 (C-O-C), 1531 (-NO_2), 890 (1,2-disubstituted benzene) and 774.3 cm^{-1} (out of plane C-H band of benzene ring), and 672 (aromatic ring C=C band). ^{1}H-NMR of PNPMCAA, In Fig.8, δ values from 7.5 to 8.1 due to present of phenyl ring and a signal of showing δ at 4.0 present of AN, δ values obtained in 2.8 to 3.7 ppm.

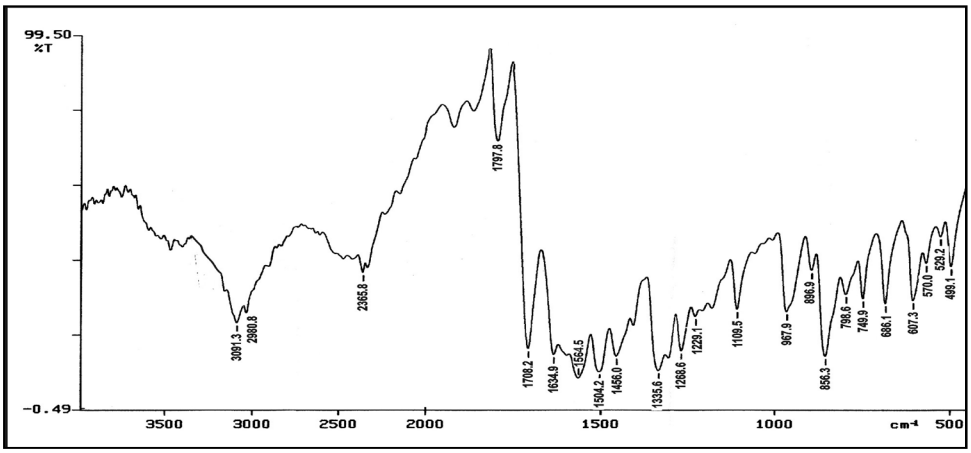

Fig.1 of PNPMI

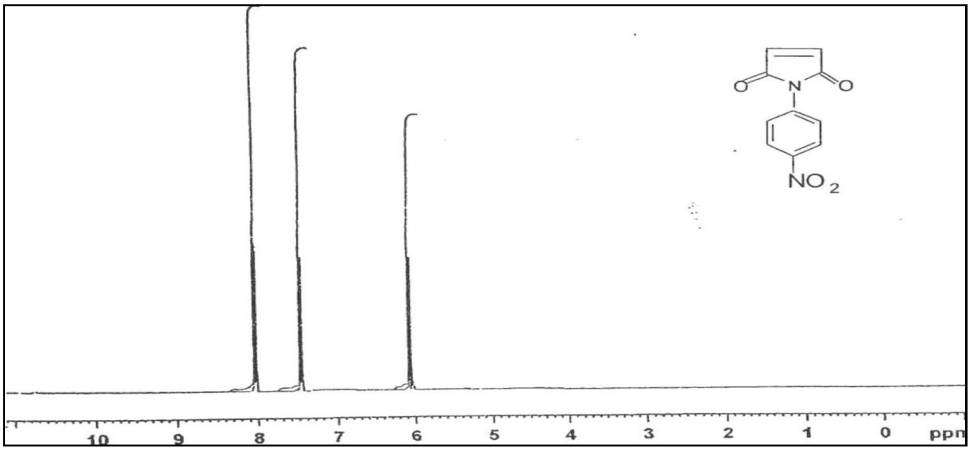

Fig. 2 For ^{1}H-NMR of PNPMI

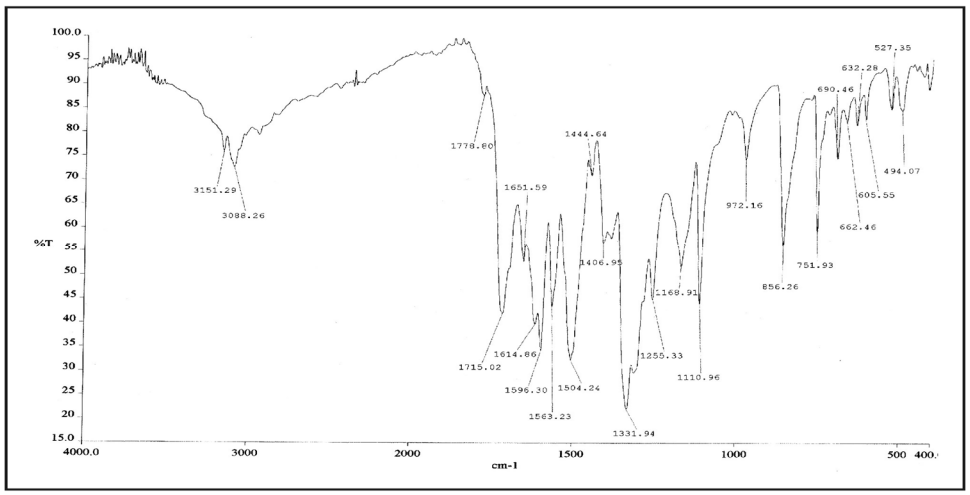

Fig. 3 FTIR spectra OF HPNPMI

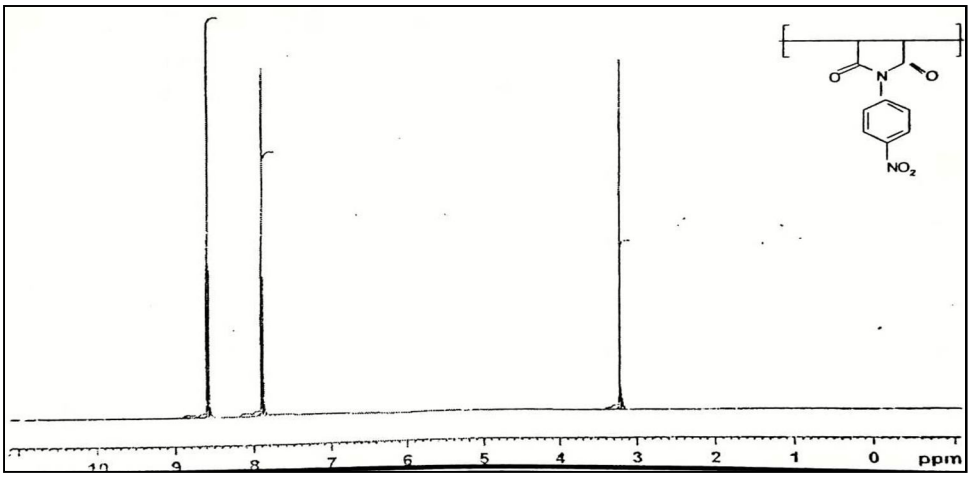

Fig. 4 For ^{1}H-NMR of HPNPMI

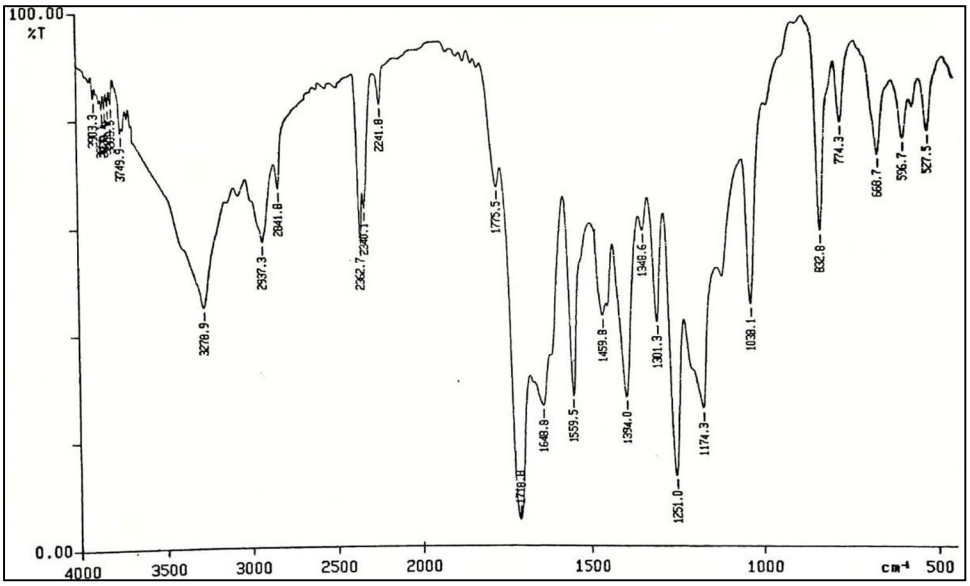

Fig. 5 FTIR spectra of PNPMCAN

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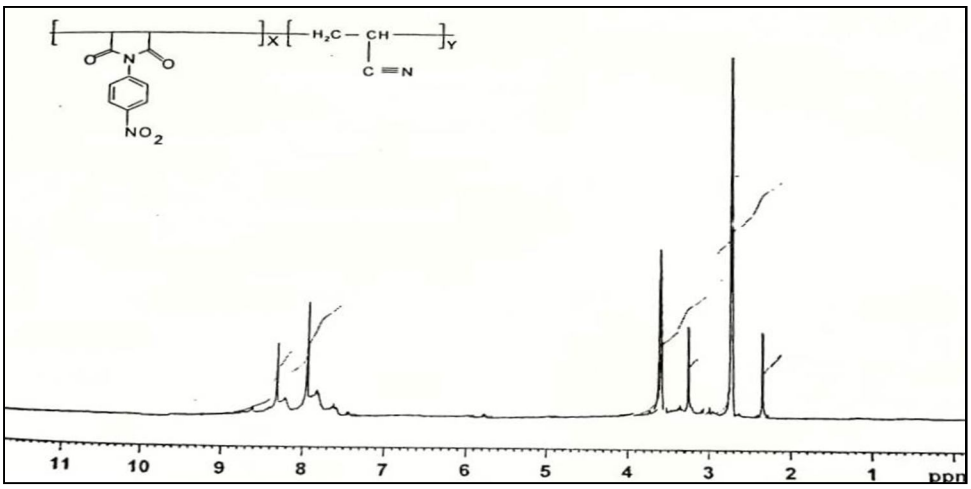

Fig.6 ^{1}H-NMR of PNPMCAN

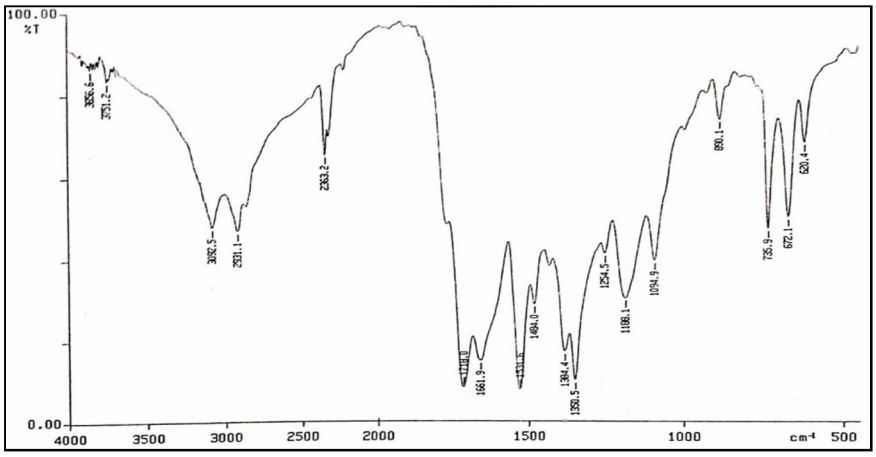

Fig. 7 FTIR spectra of PNPMCAA

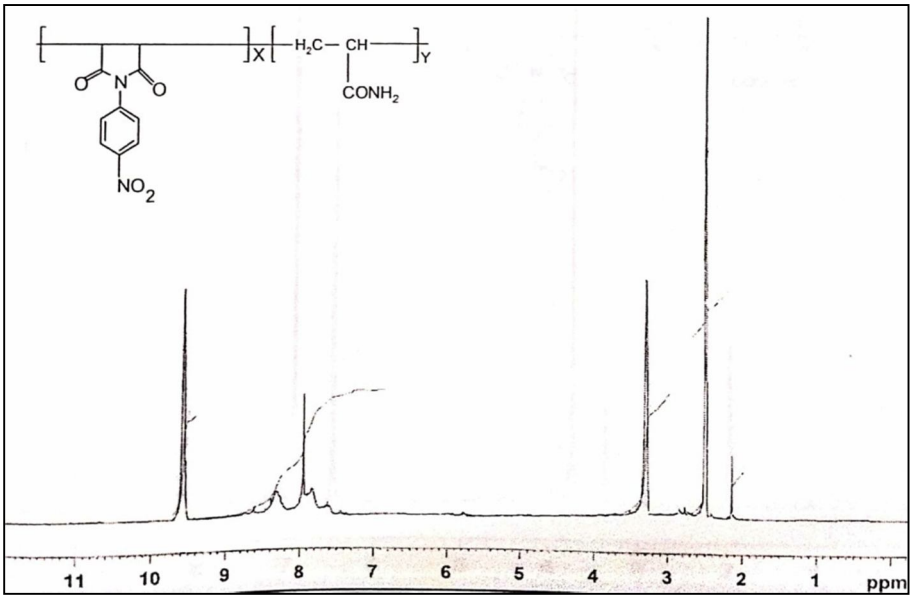

Fig. 8 ^{1}H-NMR of PNPMCAA

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E. Effect of Time on Yield

Synthesis homopolymer and copolymer and obtained percentage yield with time hours was given in table 1.

Table 1 . Percentage Yield of Homopolymer and copolymers given in table with respect to time Hours.

Polymer Code	12Hrs.	24Hr.	30Hrs.	36Hrs.	Colour
HPNPMI	18.78	33.23	38.12	40.78	Yellow
PNPMCAN	42.1	43.6	44.9	46.8	Yellow
PNPMCAA	44.0	45.2	46.1	48.2	Yellow

F. Effect of Solvent and Initiator system

Percentage yield of synthesis polymer are calculated with respect using Free radical AIBN and BPO and results are summarized in table 2.

Table 2. Shows the result of percentage yield in DMF-AIBN and DMF-BPO Solvent system

Polymer Code	Solvent	AIBN	BPO	Colour
PNPMCAN	THF	46.8	45.2	Yellow
	DMF	44.0	44.2	Yellow

Table 3. Shows the result of percentage yield in DMF-AIBN and DMF-BPO Solvent system

Polymer Code	Solvent	AIBN	BPO	Colour
PNPMCAA	THF	46.2	46.4	brown
	DMF	48.2	46.0	Yellow

Table 2 and 3 indicate that the percent yield in the AIBN-DMF system is more suitable as the comparable BPO-DMF solvent system. AIBN-DMF system gives higher yield.

G. Relation in Between Intrinsic Viscosity and Molar mass

Intrinsic viscosity determined by Ubbelohde suspended viscometer. Intrinsic viscosity depends on molecular weight as well as on the size of the polymer coil in a given solution. it is a measure of hydrodynamic volume. Molecular weight of synthesis compounds determined by the Gel Permeation Chromatography and relation find out in between molar mass and intrinsic viscosity.

Table 4. Shows the number molecular weight and Intrinsic viscosity of Synthesis polymers.

Polymer Code	Number Average(Mn)	Weight Average(Mw)	Polydispersity (Mw/Mn)	Start Mw	Intrinsic Viscosity
HPNPMI	456	519	1.138	2779	0.0423
PNPMCAN	680	1832	2.752	2660	0.238
PNPMCAA	673	1732	2.573	26657	0.268

Table 5. Typical data of Viscosity measurements of HPNPMI summarized in DMF at 30°C

Polymer code	Conc.(c) g/dl	η_{rel}	η_{sp}	η_{red}	*Intrinsic viscosity*
HPNPMI	0.1	1.0055	.0055	0.055	
	0.2	1.0138	.0138	0.069	
	0.3	1.0255	.0255	0.085	
	0.4	1.0380	.0380	0.095	0.0423
	0.5	1.0560	.0560	0.112	
	0.6	1.0732	.0732	0.122	

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Table 6. Typical data of Viscosity measurements of PNPMCAN summarized in DMF at 30°C

Polymer code	Conc.(c) g/dl	η_{rel}	η_{sp}	η_{red}	*Intrinsic viscosity*
PNPMCAN	0.1	1.0256	0.0256	0.256	0.252
	0.2	1.0522	0.0522	0.261	
	0.3	1.0792	0.0792	0.264	
	0.4	1.1084	0.1084	0.271	
	0.5	1.1365	0.1365	0.273	
	0.6	1.1662	0.1662	0.277	

Table 7. Typical data of Viscosity measurements of PNPMCAA summarized in DMF at 30°C

Polymer code	Conc.(c) g/dl	η_{rel}	η_{sp}	η_{red}	*Intrinsic viscosity*
PNPMCAA	0.1	1.0272	0.0272	0.272	0.268
	0.2	1.0554	0.0554	0.277	
	0.3	1.0855	0.0855	0.285	
	0.4	1.1152	0.1152	0.288	
	0.5	1.1455	0.1455	0.291	
	0.6	1.1776	0.1776	0.296	

Table 5,6 and 7 are showing that intrinsic viscosity of Copolymer having more than of Homopolyer it is due to present of both are unit in the structure of copolymer and molecular weight of copolymer is more than as compare to homopolymer.

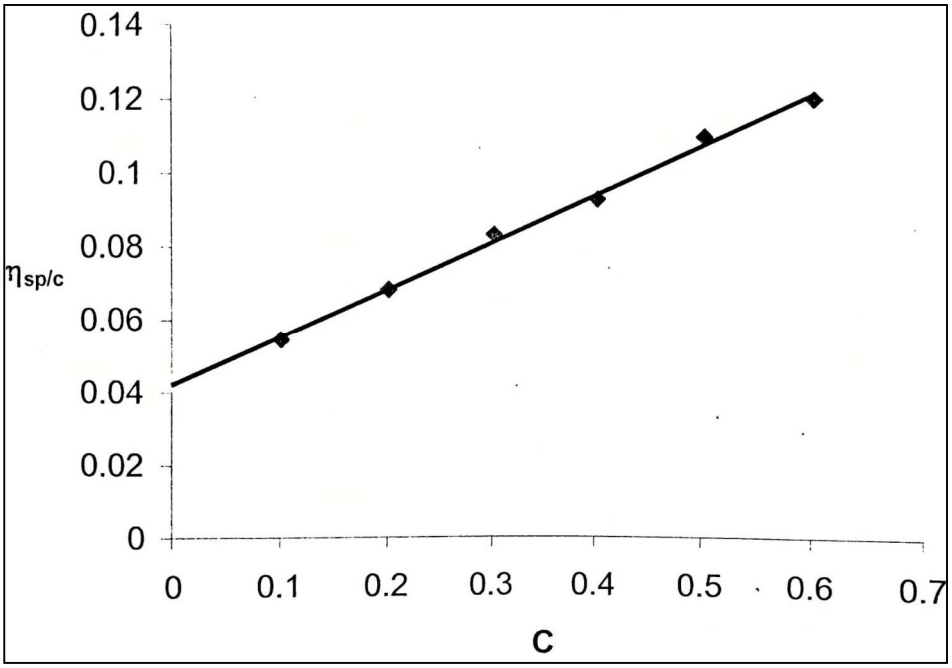

Fig. 9. for HPNPMI

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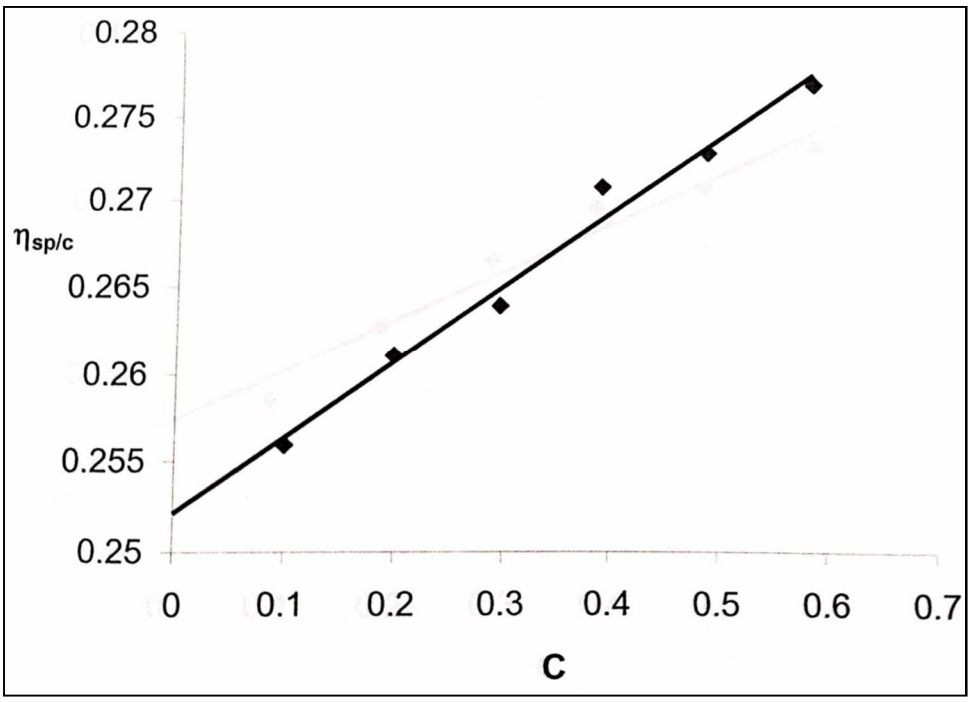

Fig. 10 .For PNPMCAN

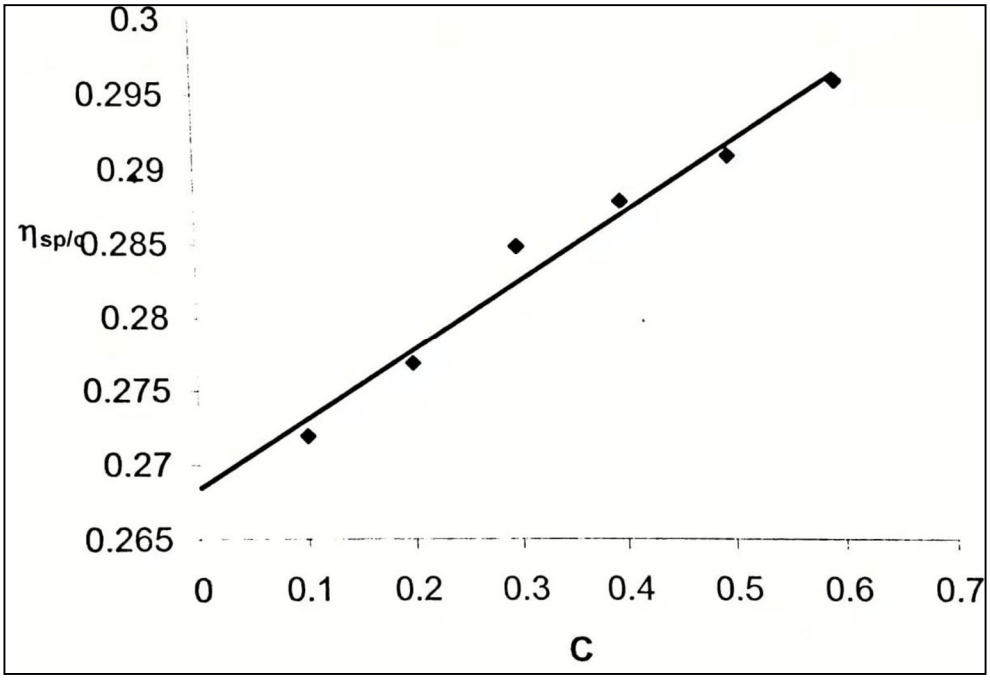

Fig. 11.For PNPMCAA

H. Solubility Behavior of Polymers

Polymers were finely grounded and polymer powder which was placed into test tube and two to three ml solvent was added and stirred at 30C. Solvents were taken as follows THF, DMF, DMSO, Benzene. Methylacetate,, 1,4Dioxane, Xylene, dichloromethane, 2Butanol,cyclohexanon,water,methanol and carbon tetrachloride. Solubility of any polymeric materials depends upon the composition like as polymer texture, temperature, pressure etc. Solubility of homopolymer is less than copolymer while always not same condition is not found in regular manner. Result of Solubility is summarized in table. 8

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Table. 8 Relative solubility of homopolymer and copolymer are summarized

Solvents	HPNPMI	PNPMCAN	PNPMCAA	Colour
Chloroform	Soluble	Soluble	S	Yellow
THF	Soluble	Soluble	S	Yellow
DMF	Soluble	Soluble	S	Yellow
DMSO	Soluble	Soluble	S	Yellow
Dioxane	Soluble	Soluble	S	Yellow
Acetone	Soluble	Soluble	S	Yellow
Ethyl acetae	Soluble	Soluble	S	Yellow
Xylene	In Soluble	Insoluble	PS	Yellow
Toluene	Soluble	PartialSoluble	PS	Yellow
Carbon tetrachloride	Soluble	PartialSoluble	PS	Yellow
Cyclohexanone	Partial Soluble	Partial Soluble	IS	Yellow
Dichloromethane	Soluble	Soluble	S	Yellow
n-Hexane	Partial Soluble	Partial Soluble	IS	Yellow
Ethanol	Soluble	Soluble	S	Yellow
Methanol	Soluble	Soluble	S	Yellow
Benzene	Soluble	Partial Soluble	S	Yellow

I. Thermal Behavior of Homo and COPOLYMER

TGA is one of the most widely used techniques for the rapid evaluation of the thermal stability and thermal degradation behaviors of various polymer [24].Thermograms of synthesis HPNPMI ,PNPMCAN and PNPMCAA are shown in Fig.12,13 and 14. Initial temperature Ti and Final temperature Tf. Degradation range shown in Table .9 and 10. The results of percentage weight loss suffered from 100 ^{o}C to 550 ^{o}C .Synthesis homopolymer and copolymer and their Thermal stability were determined by Perkin Elmer USA Pyris TGA-A Techniques and thermal degradation rate of homopolymer and copolymer are summarized in table.9 and 10.

Table 9 :Thermal degradation of Homopolymer and copolymer samples

Polymer Code	Degradation step	Ti ^{o}C	Tmax ^{o}C	Tf ^{o}C	Residue at 500 ^{o}C
PPNPMI	I	240	340	370	20.88
	II	370	410	550	
PNPMCAN	I	226	300	342	12.75
	II	342	398	540	
PNPMCAA	I	200	275	342	6.85
	II	342	420	550	

Table 10 .Percentage weight loss of Homopolymer and copolymer at various temperature.

Polymer Code	200 ^{o}C	300 ^{o}C	400 ^{o}C	500 ^{o}C	550 ^{o}C
PPNPMI	-0.8	-20.8	-72.8	-79.1	-79.2
PNPMCAN	-2.82	-50.28	-80.88	-87.25	-88.29
PNPMCAA	-0.91	-7.89	-48.75	-84.79	-92.95

From the table 9 and 10 gives the comparative study because percentage degradation in homopolymer shows very less, while percentage degradation in copolymer shows very high it is due to homopolymer having only one unit while copolymer having two units and percentage degradation of the thermal curves are shows in fig.12,13 and 14.

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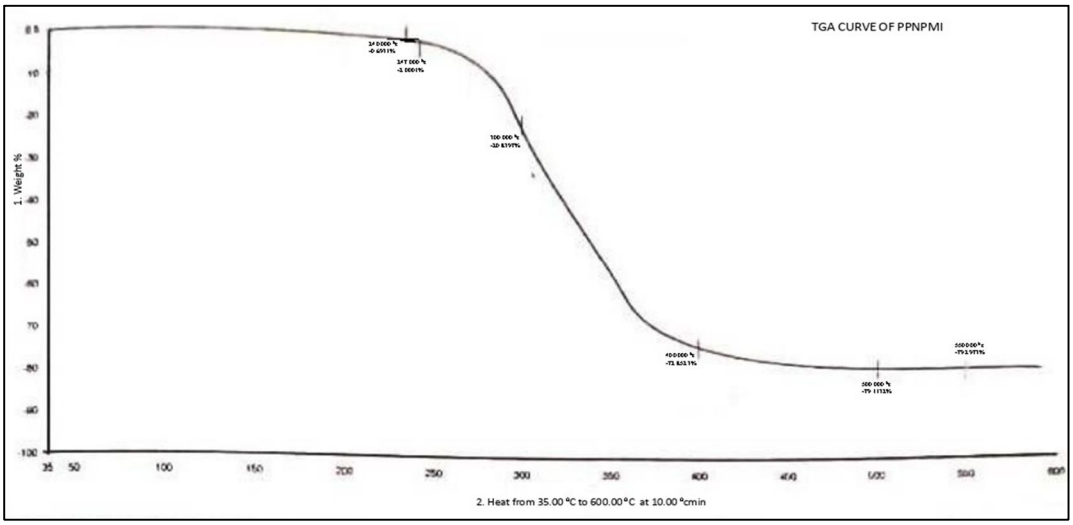

Fig. 12 , Thermogram Of PPNPMI

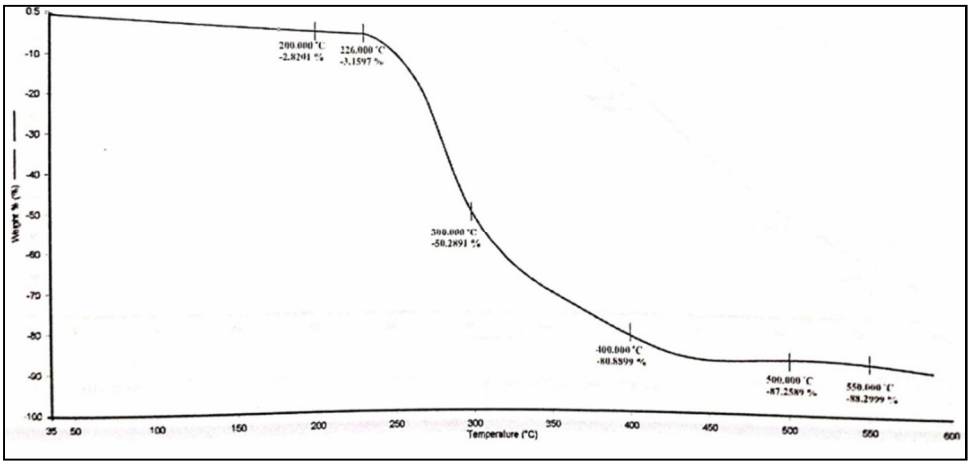

Fig.13 Thermograms for PNPMCAN

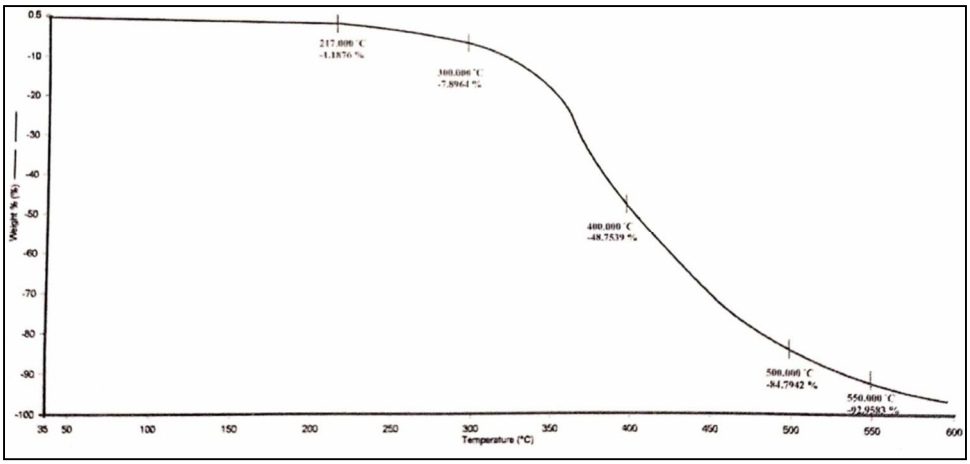

Fig. 14. Thermograms for PNPMCAN

IV. CONCLUSION

Synthesis homopolymer and copolymer shows the excellent solubility in THF,DMF ,DMSO, Chloroform .Ethyl acetate and solubility of any polymeric materials depends on the composition like as texture and length ,molecular weight, Intrinsic viscosity. It is observed that polymerization reactions completed via radical polymerization this is approved by the Polydispersity index. Copolymer shows highly molecular weight more than of homopolymer it is due to copolymer having both are unit. Weight average and number average molecular weight determined by the Gel permeation chromatography. Polymers exhibit highly thermal stability due to present of imide ring and Homopolymer shows highly thermal stability than copolymers because homopolymer contain only single unit in their backbones.

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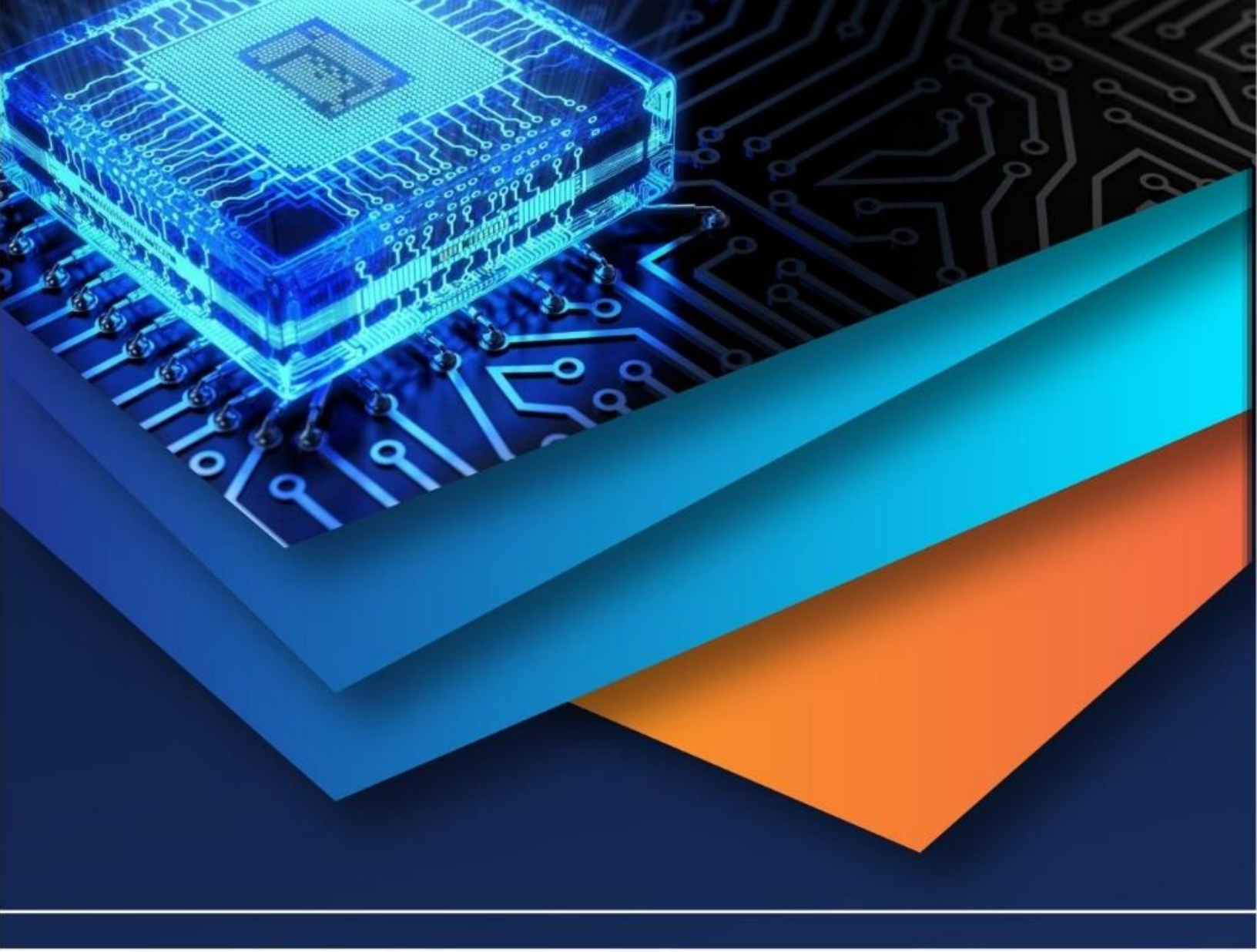

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