

Comparison of Thermal Degradation, WAXRD Studies and SEM of Renewable Resources Based Interpenetrating Polymer Networks

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Abstract: Recently bio-based polymers have received great importance due to their eco-friendly properties towards environment. As a substitute of conventional reinforcing synthetic polymer, bio-based polymers were from renewable resources like cardanol, i.e., the meta-substituted phenolic compound the chief product of CNSL. A number of IPNs have been synthesized by condensing di-azotised-4-amino benzoic acid and 3-amino benzoic acid cardanol dye with polyurethane of soybean oil. The IPNs produced have been characterized by FTIR, TGA, DSC (Differential scanning calorimetry). Elemental analysis and WAXRD studies. Thermal stability of IPNs has been studied by both TGA and DSC which provides heat capacity and kinetic parameters (order of reaction and activation energy) by Freeman Anderson method of the sample.

Keywords: Cardanol based dye, thermal stability, crystallinity, activation energy, interpenetrating polymer network.

I. Introduction

A survey of literature reveals that bio based polymer materials widely used in paints, varnishes, coatings, adhesives. These IPNs has been synthesized by diazotised cardanol with Pus of soybean oil. As both the starting materials of the new polymers are natural resources, hence polymer chemists have turned their attention to this synthesis. This paper aims to present a highly cross-linked polymer from agricultural products which have high temperature resistance. The structural confirmation of the polymer has been established by FT-IR and WAXRD studies. Thermal resistance is studied by TGA and DSC.

Experimental Section

Materials:

- Refined soybean oil obtained from market.
- Cardanol was obtained from fractional distillation of CNSL liquid, a by-product of Sathya cashew chemical pvt. Ltd., Chennai.
- NaOH, MEK, EGDM, NaNO₂, HCl, PbO, etc. Were obtained from M/S BDH. Ltd. (INDIA)
- TDI, DPMDI, 3-amino benzoic acid and 4-amino benzoic acid from E-merk (GERMANY). All chemicals were used as received

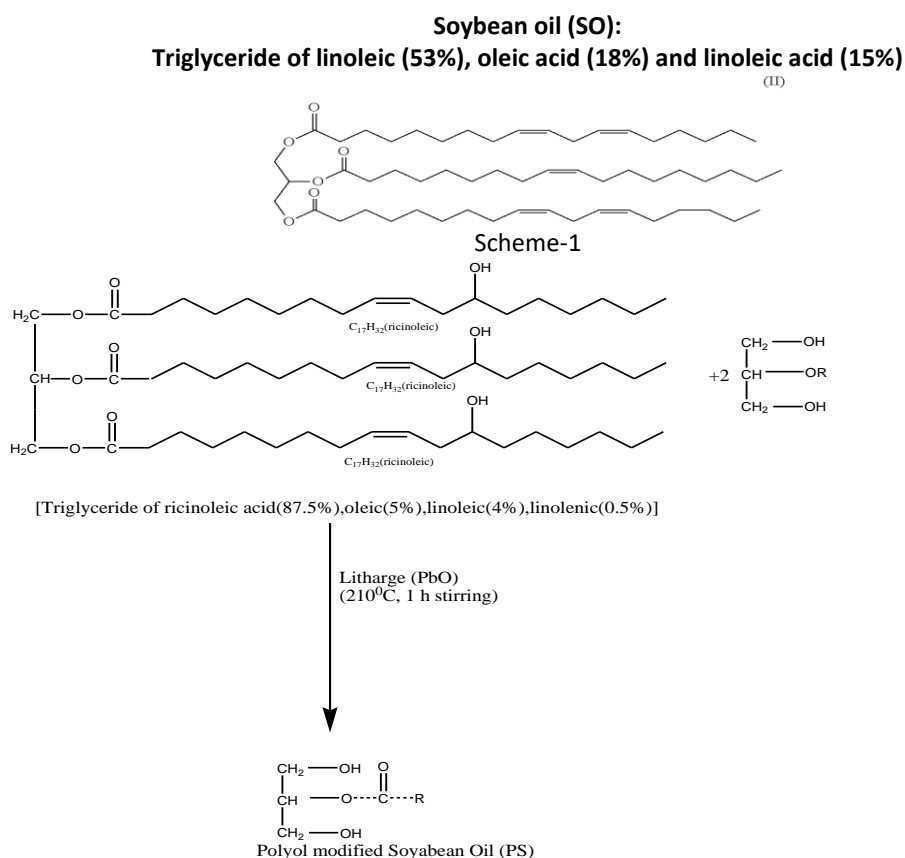
Methods:

1. **Spectroscopic Analysis- FTIR (Fourier transform infrared):** FTIR spectra of the prepared IPNs samples have been recorded on FTIR Spectrophotometer by Thermo Electric Corporation, USA, and Model: Nicolet 670 FT-IR using KBr pallette in the wavelength range of 500cm⁻¹ to 4000cm⁻¹.
2. **Thermal Analysis (DSC and TGA):** DSC and TGA of all IPNs have been performed by use of a Universal v4.5A.TA instrument (Model SDT Q 600 V20.9 Build 20) at a heating rate of 10⁰c/minute.
3. **Morphology study (SEM):** Morphology of samples has been studied by JOEL scanning electron microscope (SEM) Model JSM 500. For this the fractured samples have been coated with a thin layer of gold- platinum alloy by sputtering to provide conductive surface.
4. **Element detection:** IPNs are heated for 30 seconds in different scale count and the percentage of Carbon, Nitrogen and Oxygen atoms are given in the graphs.
5. **Study of Crystallinity- Wide Angle X-ray diffraction study (WAXRD):** X-ray diffraction pattern of polymer samples have been collected using a panalytical x'pert pro Θ/Θ goniometer with Cu - K α radiation.
6. **Test for biodegradability:** The environmental resistance of the IPNs samples was carried out using soil burial test.

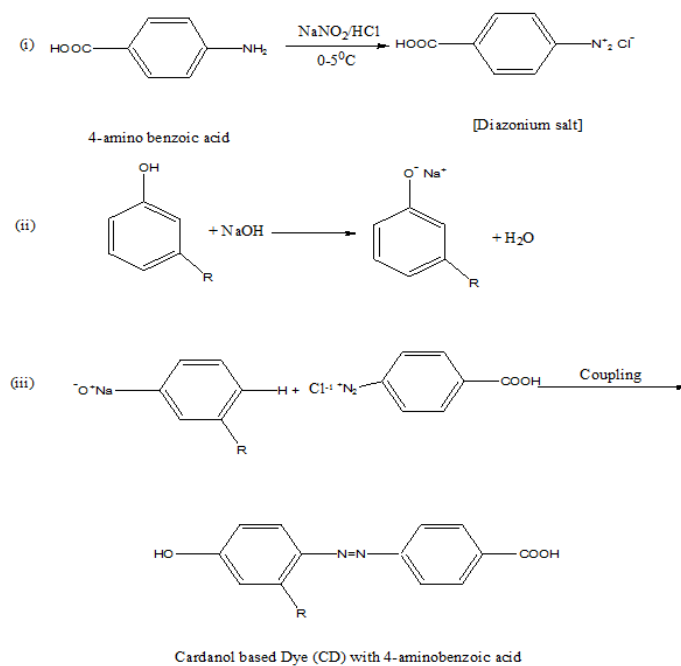
Experimental:-

- i. Preparation of dye monomer-** 6.85g of 4- amino benzoic acid was dissolved in 25ml of conc. HCl acid and 25ml of water was added to it. The solution was cooled 0⁰-5⁰C, and then a cold solution of sodium nitrite (4g in 20ml of water) was added to it slowly with stirring for 3-4 minutes. A cold solution of cardanol (15g in 45ml) of 10% NaOH solution was prepared. Then cold diazonium solution was added slowly to the alkaline cardanol solution with stirring. A brown colour semi liquid dye was formed. The dye was separated by a separating funnel.
- ii. Preparation of Mixed Ester Polyol (MEP) from Soybean Oil(SO):** Refined soybean oil (350ml) was heated at 250⁰ C in an inert Nitrogen atmosphere taken in three naked flask fitted with a thermometer reflux condensers and a stirrer. At this temperature litharge (0.168g) and glycol(80ml) were added to the reaction mixture with constant stirring. The temperature was maintained at 210⁰C until one volume of reaction mixture gave a clear solution in same volume methanol. At this stage the contents were cooled to obtain MEP.
- iii. Synthesis of Polyurethane (PU):** 1 mole of MEP was added to 1.6 mole of TDI to maintain NCO/OH ratio at 1.6. The reaction was carried out at 75⁰C with continuous stirring for one hour until a viscous pale yellow colour PU is separated out. The same process was repeated with different NCO/OH ratio (1.2, 1.6, and 2.0) and with other diisocyanate DPMDI PUs was produced.
- iv. Synthesis of IPNs:** The mixture of PU and diazotised cardanol (with different PU/monomer ratio i.e., (75:25, 50:50, 65:35) and solvent (MEK) were taken in small beaker. Then 5ml of 10% EGDM along with 20mg of Benzoyl peroxide (BPO) were added to each mixture. The total mixture was stirred for 15 minutes with magnetic stirrer in cold to obtain a homogenous solution. Then the temperature is raised to 75⁰C and stirred until a thick solution was formed. Then the viscous mass was poured into a petridish in hot condition and kept in an oven at 75⁰C for 24 hour. The feed composition data of the different IPNs are given in table-1

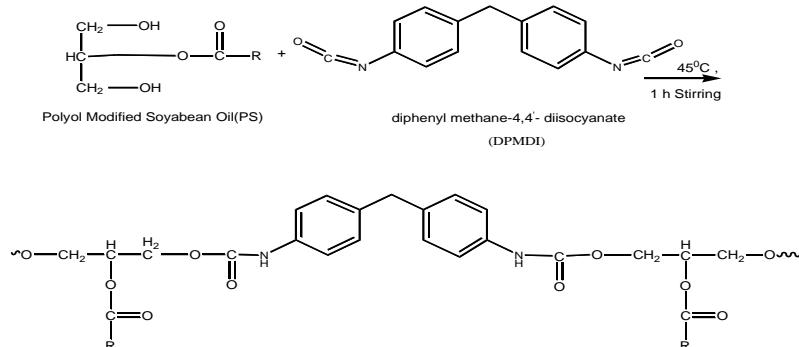
The thin film thus obtained was cooled and removed from the petridish with a sharp blade and sent for characterisation to the Central Instrumentation Facility Pondicherry University, Puducherry, pin- 605014.



Scheme – 2

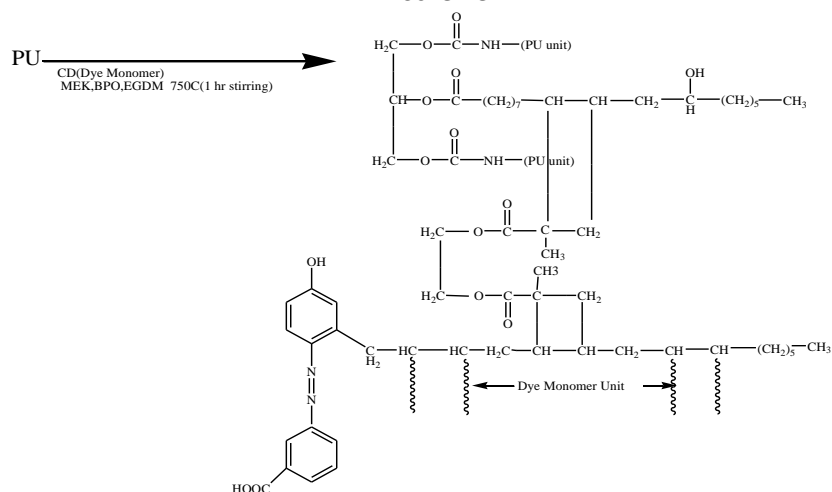


Scheme – 3



(POLYURATHANE (PU))

Scheme – 4



(INTERPENETRATING POLYMER NETWORK FROM NATURAL RESOURCES)

Analysis of the Sample:

The FTIR spectra of the IPNs are presented in figs 1-a to 1-d

FTIR OF IPN 27

The characteristic absorption of IPN-27 corresponding to O-H stretching of >OH groups shifted to lower value by hydrogen bonding at 3858.6 cm⁻¹. N-H stretching of >NH group at 3444.5 cm⁻¹, C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2861.9 cm⁻¹ and 2747.9 cm⁻¹, N≡C stretching of -N=C=O group for the isocyanate terminating PU unit at 2319.9 cm⁻¹, C=O stretching of urethane linkage at 1660.3 cm⁻¹, N=N stretching of azo group at 1547.6 cm⁻¹, C-O bending at 1029.3 cm⁻¹, C-C stretching of aromatic rings at 1398.8 cm⁻¹, out of plane C-H bending at 806.6 cm⁻¹ and out of plane C-C bending at 691.8 cm⁻¹ and 551.6 cm⁻¹ were observed.

FTIR OF IPN 32

The characteristic absorption of IPN-32 corresponding to -OH stretching of >OH groups shifted to lower value by hydrogen bonding at 3446.3 cm⁻¹. The C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2797.6 cm⁻¹ and 2921.6 cm⁻¹, N≡C stretching of -N=C=O group for the isocyanate terminating PU unit at 2364.4 cm⁻¹, C=O stretching of urethane linkage at 1720.4 cm⁻¹ and 1661.0 cm⁻¹, N=N stretching of azo group at 1581.2 cm⁻¹, N=N stretching of aromatic rings at 1581.2 cm⁻¹, C-O bending at 1161.8 cm⁻¹, C-C stretching of aromatic rings at 1259.2 cm⁻¹, C=C stretching of aromatic rings at 1392.6 cm⁻¹, C-O stretching of ester at 1161.8 cm⁻¹, out of plane C-H bending at 703.5 cm⁻¹ and out of plane C-C bending at 516.4 cm⁻¹ were observed.

FTIR OF IPN 35

The characteristic absorption of IPN-35 corresponding to -OH stretching of >OH groups shifted to lower value by hydrogen bonding at 3606.2 cm⁻¹. N-H stretching of >NH group at 3470.4 cm⁻¹, C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2921.0 cm⁻¹ and 2861.0 cm⁻¹, N≡C stretching of -N=C=O group for the isocyanate terminating PU unit at 2773.8 cm⁻¹ and 2322.6 cm⁻¹, C=O stretching of urethane linkage at 1661.4 cm⁻¹, C-O bending at 1164.2 cm⁻¹, C=C stretching at 1540.2 cm⁻¹, C=C stretching of aromatic rings at 1540.2 cm⁻¹, C-O stretching of ester at 1161.0 cm⁻¹, -OH bending of -COOH group at 1310.3 cm⁻¹, out of plane C-H bending at 869.6 cm⁻¹ and out of plane C-C bending at 713.8 cm⁻¹ were observed.

FTIR OF IPN 36

The characteristic absorption of IPN-36 corresponding to -OH stretching of >OH groups shifted to lower value by hydrogen bonding at 3855.4 cm⁻¹ and 3734.8 cm⁻¹. N-H stretching of >NH group at 3395.3 cm⁻¹, C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2920.4 cm⁻¹ and 2859.7 cm⁻¹, N≡C stretching of -N=C=O group for the isocyanate terminating PU unit at 2773.1 cm⁻¹, 2545.7 cm⁻¹, and 2362.8 cm⁻¹, C=O stretching of urethane linkage at 1727.6 cm⁻¹, N=N stretching of azo group at 1579.3 cm⁻¹, C-C stretching of aromatic rings at 1404.7 cm⁻¹, C=C stretching of aromatic rings at 1530.7 cm⁻¹, C-O stretching of ester at 1161.9 cm⁻¹, -OH bending of -COOH group at 1306.3 cm⁻¹, out of plane C-H bending at 811.5 cm⁻¹ and out of plane C-C bending of p-substituted benzene rings at 644 cm⁻¹ and 483 cm⁻¹ were observed.

CONCLUSION

From the fig.1a to 1d correlation with the peak of the authentic compound helps to identify the polymer sample. The -OH stretching shifted to lower value by hydrogen bond at 3607 and 3897 cm⁻¹. The N-H stretching of >NH₂ group at 3333.5 cm⁻¹, C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2926.4 cm⁻¹ and 2857.7 cm⁻¹, N≡C stretching of -N=C=O group for the isocyanate terminating PU unit at 2341 cm⁻¹, C=O stretching of urethane linkage at 1660-1728 cm⁻¹, N=N stretching of azo group at 1590 cm⁻¹ to 1620 cm⁻¹, C-C stretching of aromatic rings at 1399 cm⁻¹, C=C stretching of aromatic rings at 1518 cm⁻¹, -OH bending of -COOH group at 1120-1240 cm⁻¹, out of plane C-H bending at 784 cm⁻¹ and out of plane C-C bending of p-substituted benzene rings at 863.1 cm⁻¹.

D.Sc. (Differential Scanning Calorimetric Analysis):

This method is used to characterize thermo physical properties of a polymer sample like melting point, heat of melting, percentage of crystallinity, T_g or softening of the polymer. This method also helps us to observe phase transition.

Heat capacity C_p = (Q/m) ΔT

- Q: Heat added
- m: mass of material
- ΔT = (T_s - T_r) = Change in temperature

ΔT is zero for no reaction

Heat of fusion = Q/m

$$C_p = \frac{d}{dT/dt} \times \frac{1}{m}$$

$$\text{Percentage of crystallinity} = \frac{\Delta H_m - \Delta H_c}{\Delta H_m} \times 100$$

Conclusion

From the table-2 it is evident that IPN₂₇, IPN₃₅, IPN₃₆ are more thermally stable than IPN₃₂ which has T_m value 420.6 and lowest molar ratio. While other three IPNs are comparatively more thermally stable. IPN₃₂ has more T_g value than IPN₂₇, IPN₃₅, IPN₃₆ due to lower PU content and NCO/OH ratio (1.6).

Thermogravimetric analysis

Thermogravimetric analysis of IPNs was carried out as a function of weight loss verses temperature. Thermograms are interpreted and analysed to obtain information about the percentage of weight loss at different temperatures from which kinetic parameters has been calculated in

Table-3a following Freeman-Anderson method and are shown in graphs named Fig 3a to 3h

Conclusion:

IPN₃₂ is less stable at higher temperature than IPN₂₇, IPN₃₅, IPN₃₆. All the three IPNs are stable upto 700⁰C polymer chain breaks nearly about 400⁰C to 450⁰C.

IPN₂₇, IPN₃₅, IPN₃₆ all have same NCO/OH molar ratio (i.e.2.0) are more stable than IPN₃₂ lower NCO/OH ratio (1.6).

Kinetic parameters of IPNs by Freeman-Anderson method have been calculated as per the following equation and are shown in table 3b.

$$\Delta \log \left(-\frac{dw}{dt} \right) = n \Delta \log w - \frac{E_a}{2.303R} \Delta \left(\frac{1}{T} \right)$$

Where n= order of reaction

E_a= Activation Energy

These values are determined from the plot of $\Delta \log \left(-\frac{dw}{dt} \right)$ vs $\Delta \log w$. The slope gives us order of reaction n and intercept is related to activation energy (E_a) which is given by E_a which is given by

$$E_a = \frac{-\text{Intercept} \times 2.303 R}{\Delta \left(\frac{1}{T} \right)}$$

Element detection:

IPNs are heated for 30 seconds in different scale count and the percentages of Carbon, Nitrogen and Oxygen atoms were found which are given in graphs named as Fig 5a,5b,5c,5d and table- 4a to 4k respectively.

4. Wide angle X ray diffraction (WAXRD)

This is a very good technique to analyse a polymer sample to know the percent of crystallinity. It is a non destructive method of characterisation of solid polymers. The samples have been scanned in a wide angle X ray goniometer and the scattering intensity is plotted as a function of 2 Θ angle (2 Θ is larger than 5⁰) as shown in fig- 4a,4b,4c,4d and table-5a,5b,5c,5d respectively.

Conclusion

From the above fact it is clear that IPN₂₇ is more crystalline than IPN₃₂, IPN₃₅, IPN₃₆ all the IPNs prepared is a mixture of amorphous and crystalline substance are known as polymer blend. Each sample has a unique pattern of d-spacing which is the figure print of that sample. The percentage of crystallinity increases with more PU content and decreases with more dye.

SEM (scanning electron microscope) Study

This technique has been used to study the morphology of polymer blends or samples. Morphology of polymer blends or samples consists of two distinct phases which was very clear in photos(fig- Fig 6a to 6h). Interpenetration of two phases i.e. crystalline and amorphous are also clearly seen in the photos named as Fig 6a to 6h respectively.

CONCLUSION

IPN – 32 and IPN-35 exhibited greater degree of interpenetration, phase mixing and good morphology as compared to IPN-36 and IPN-27. This morphology is expected due to the ratio of PU and CD. This study shows complex phase behaviour of IPNs. Increase in PU content the heterogeneity increases and morphology changes from continuous to discontinuous phase.

BIODEGRADABILITY:

The IPN samples synthesized were tested for environmental resistance by use of soil burial test. The samples were buried in soil for sixty days. The samples were removed from the soil once in fifteen days to access the changes in their weight loss, mechanical strength and surface damage if any.

II. Conclusion

From this observation(table-6) it is seen that these samples are not biodegradable, only a small amount of it decomposes with the bacteria and virus present in the soil which is very negligible.

Acknowledgement

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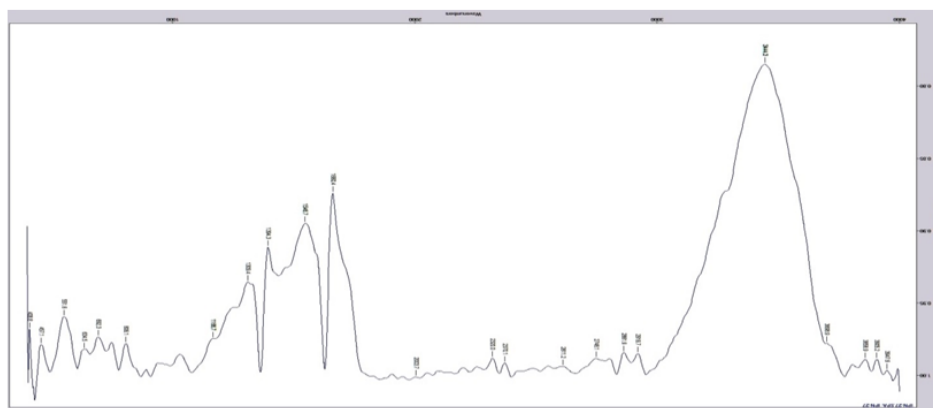


Fig 1a: FTIR OF IPN 27

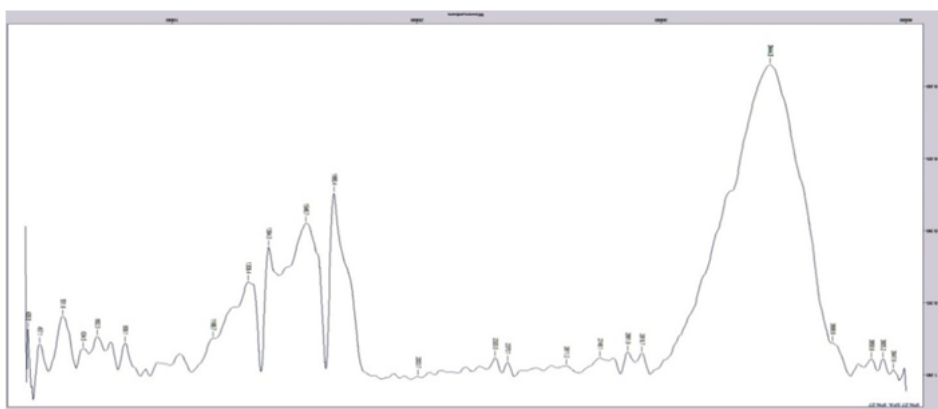


Fig 1b: FTIR OF IPN 32

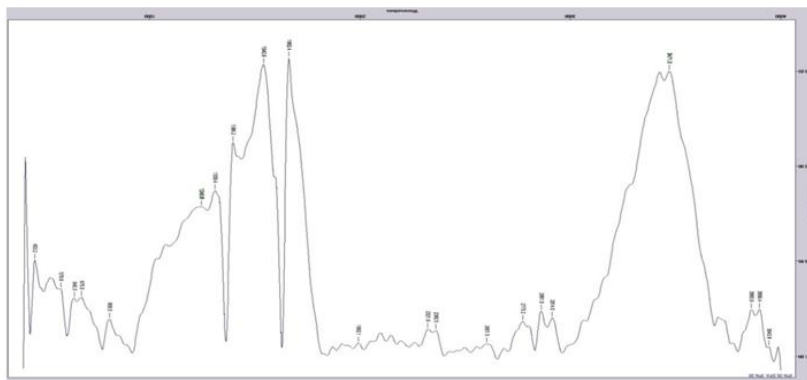


Fig 1c: FTIR OF IPN 35

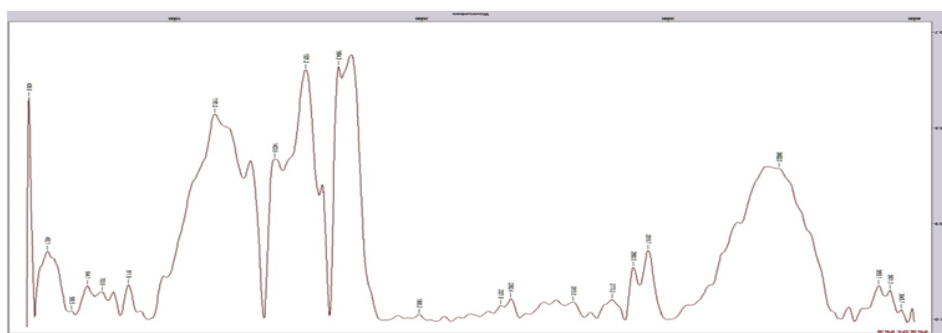


Fig 1d: FTIR OF IPN 36

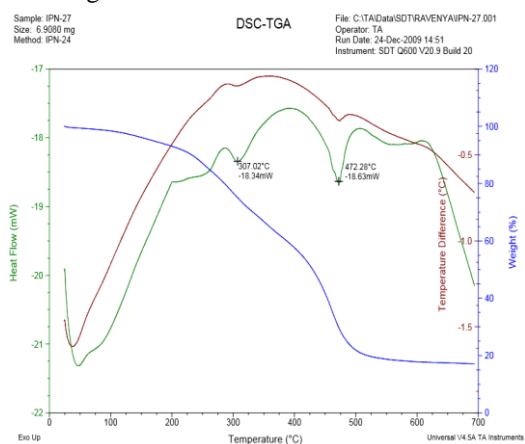


Fig 2a - DSC TGA OF IPN 27

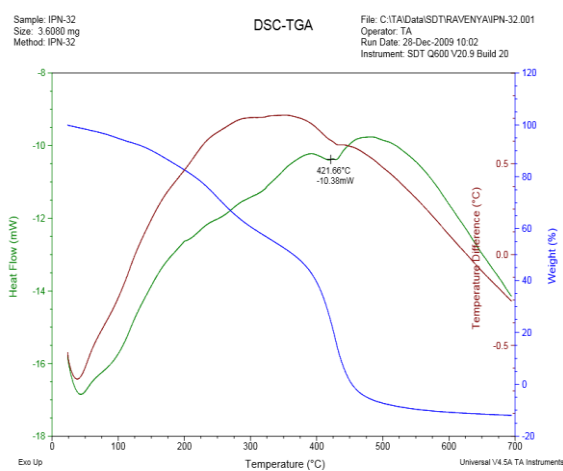


Fig 2b - DSC TGA OF IPN 32

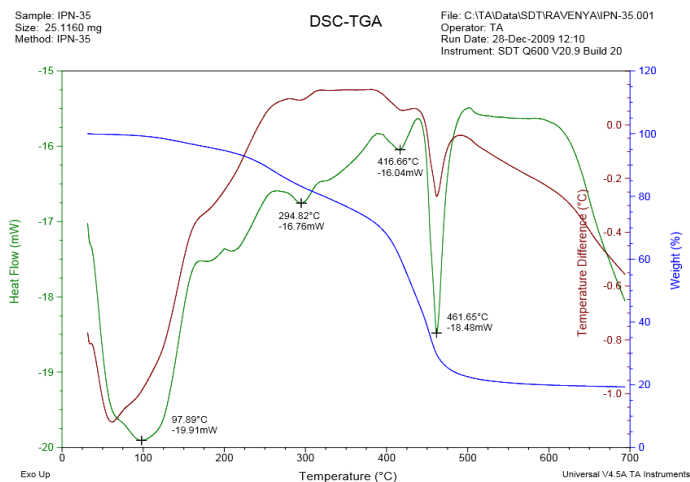


Fig 2c - DSC OF IPN 35

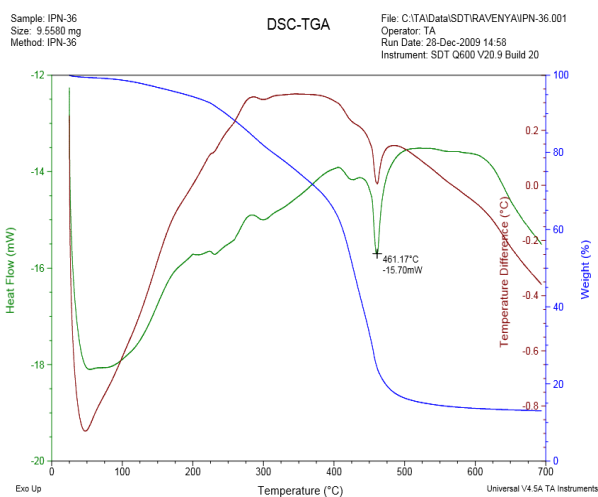


Fig 2d - DSC OF IPN 36

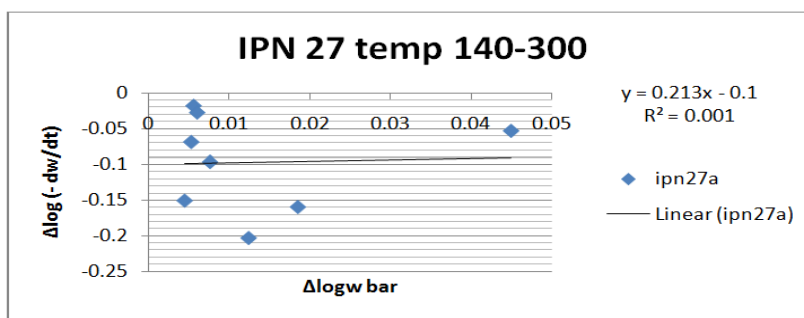


Fig 3a –Freeman-Anderson Plot of IPN27

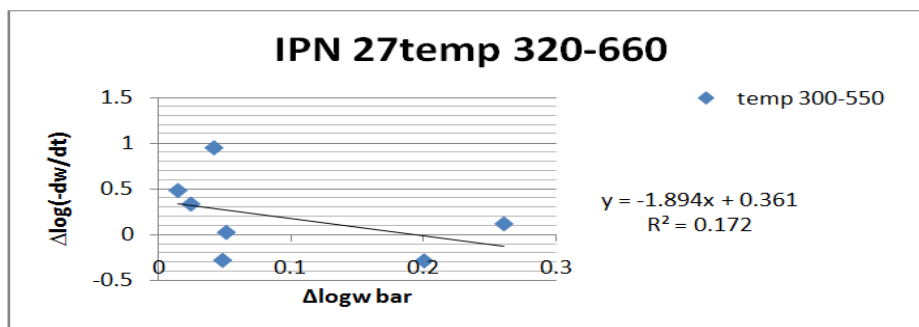


Fig 3b –Freeman-Anderson Plot of IPN27

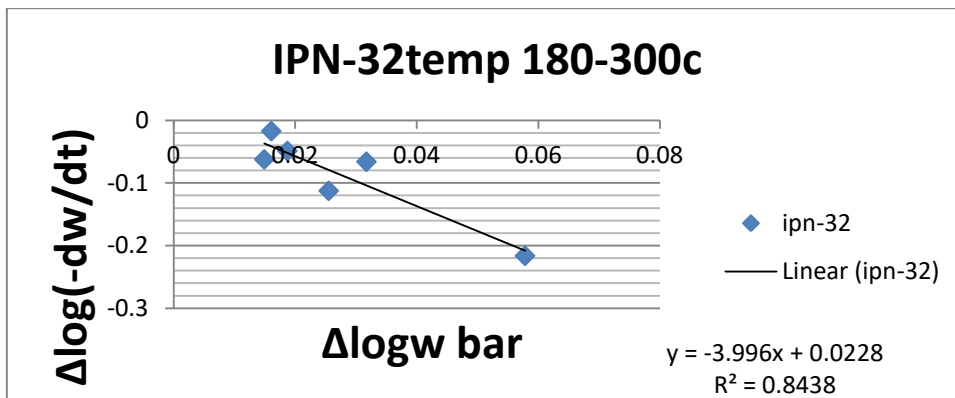


Fig 3c –Freeman-Anderson Plot of IPN32

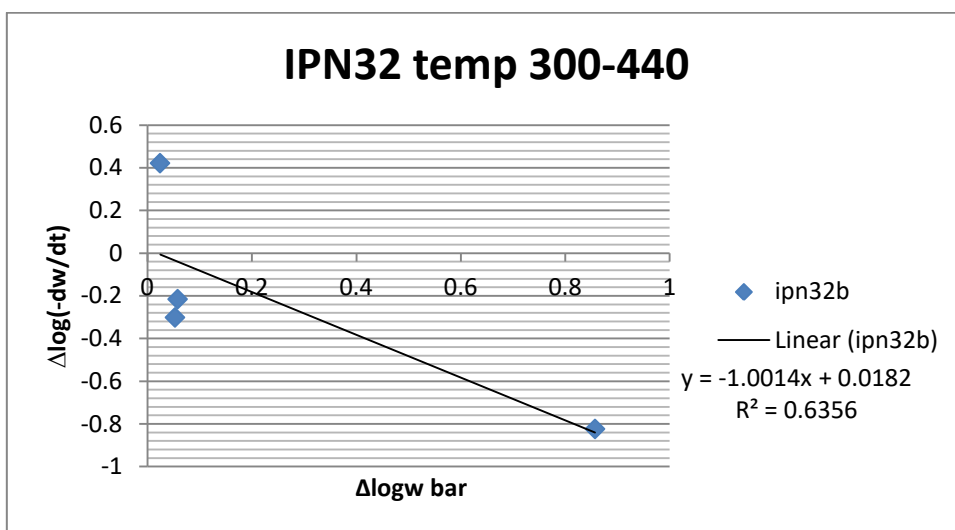


Fig 3d –Freeman-Anderson Plot of IPN32

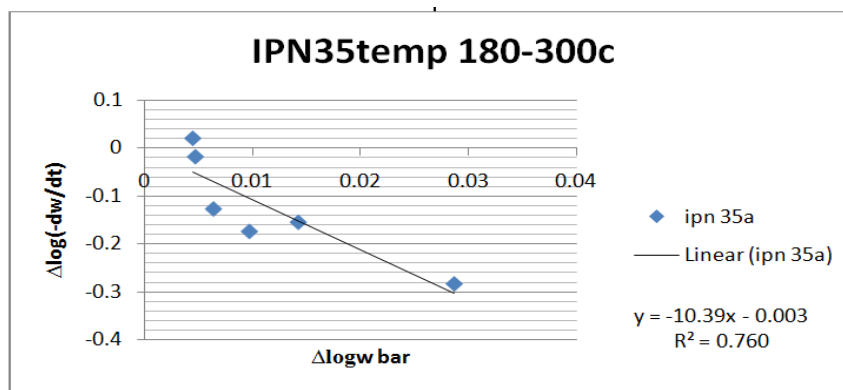


Fig 3e –Freeman-Anderson Plot of IPN35

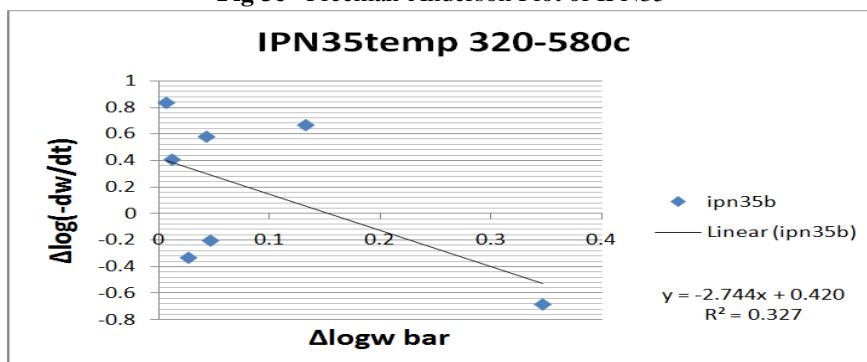


Fig 3f –Freeman-Anderson Plot of IPN35

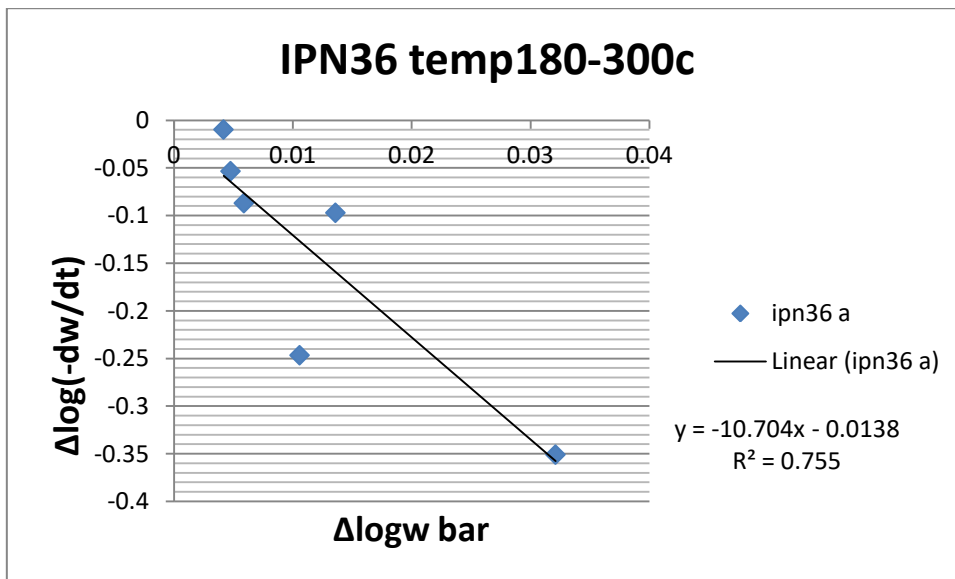


Fig 3g –Freeman-Anderson Plot of IPN36

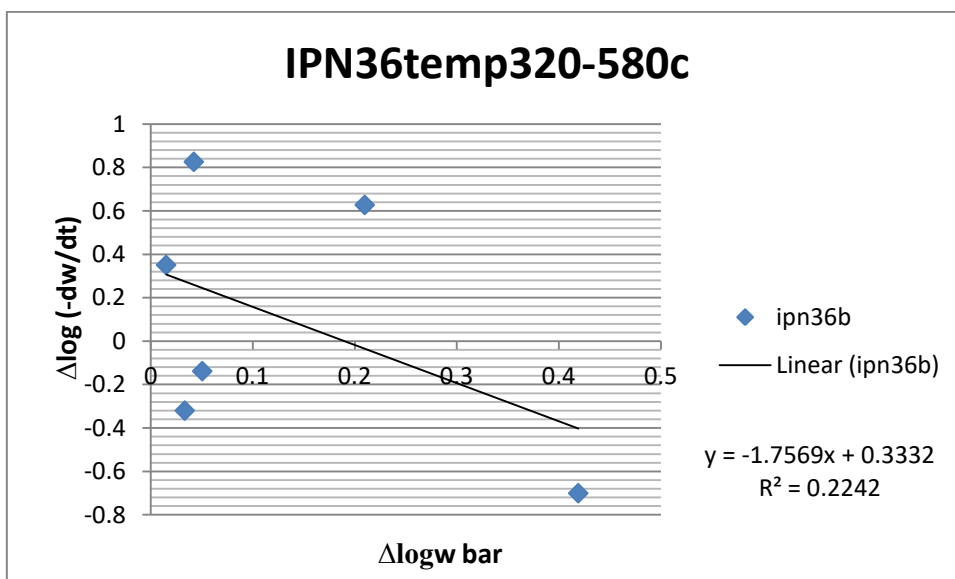


Fig 3h –Freeman-Anderson Plot of IPN 36

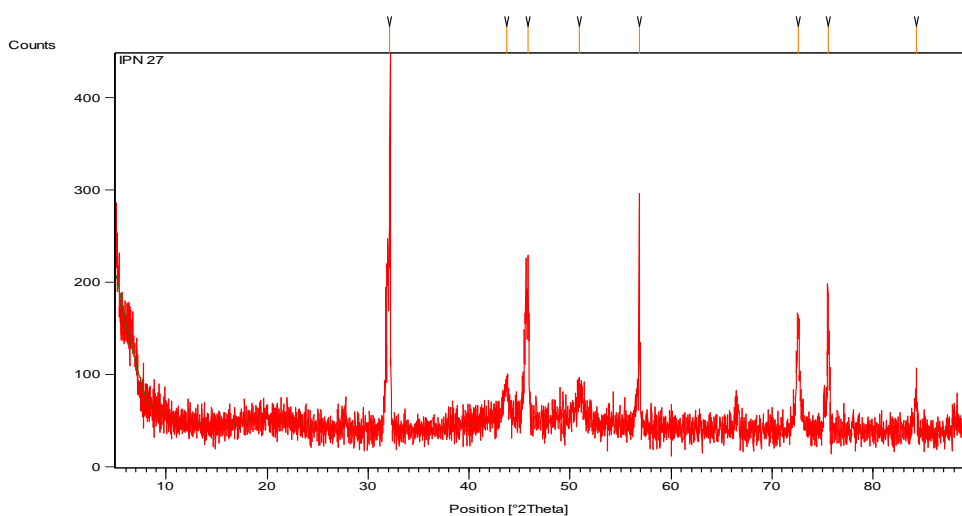


Fig 4a –WAXRD OF IPN 27

Peak List: (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
32.1472	296.40	0.1440	2.78215	100.00
43.6773	39.96	0.5760	2.07073	13.48
45.8599	152.13	0.1920	1.97713	51.33
50.9136	31.27	1.1520	1.79210	10.55
56.8643	145.80	0.1920	1.61787	49.19
72.5808	112.13	0.2880	1.30145	37.83
75.5410	139.77	0.1920	1.25763	47.15
84.2679	39.83	0.2880	1.14822	13.44

Table 4a

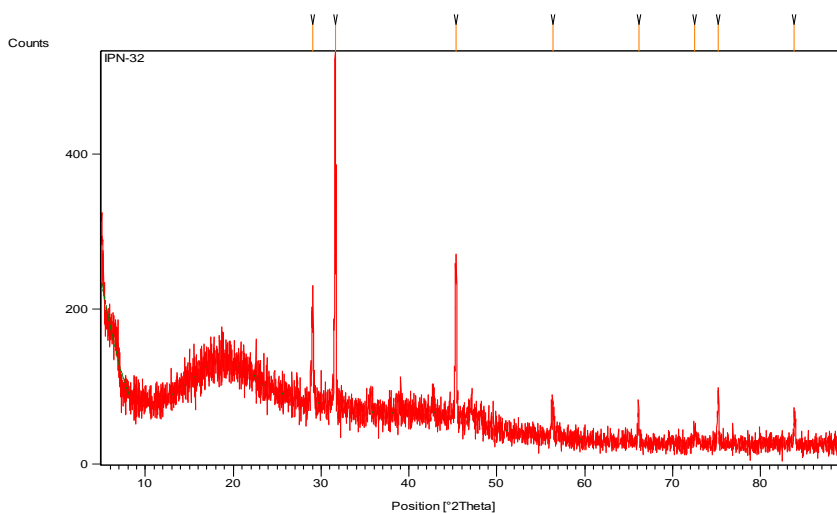


Fig 4b –WAXRD OF IPN 32

Peak List: (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
29.0285	115.74	0.1920	3.07356	34.09
31.6297	339.47	0.1920	2.82648	100.00
45.3866	207.38	0.1920	1.99664	61.09
56.3433	40.44	0.2880	1.63159	11.91
66.1179	43.32	0.1920	1.41209	12.76
72.5183	11.55	1.1520	1.30242	3.40
75.1681	65.91	0.1920	1.26294	19.42
83.8281	26.47	0.5760	1.15312	7.80

Table 4b

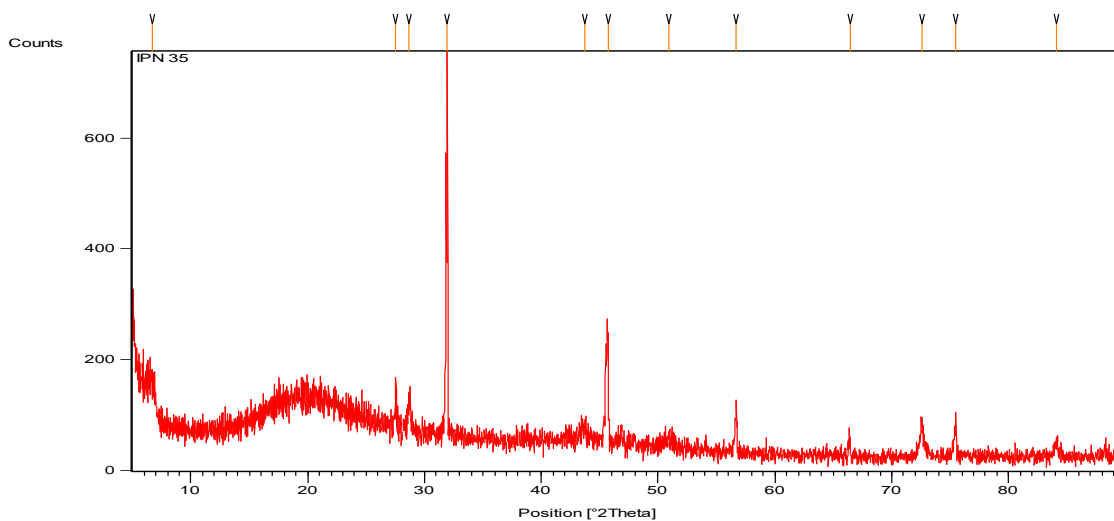


Fig 4c –WAXRD OF IPN 35

Peak List: (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
6.6666	90.67	0.7680	13.24802	19.91
27.5257	37.27	0.2880	3.23786	8.18
28.6765	56.19	0.2880	3.11048	12.34
31.9249	455.32	0.2400	2.80101	100.00
43.7147	23.36	1.1520	2.06904	5.13
45.7059	198.80	0.1440	1.98343	43.66
50.8830	16.76	1.1520	1.79311	3.68
56.7027	80.56	0.1920	1.62210	17.69
66.4155	41.35	0.2880	1.40648	9.08
72.5588	64.51	0.2880	1.30179	14.17
75.4177	52.92	0.2880	1.25938	11.62
84.1301	32.61	0.2880	1.14974	7.16

Table 4c

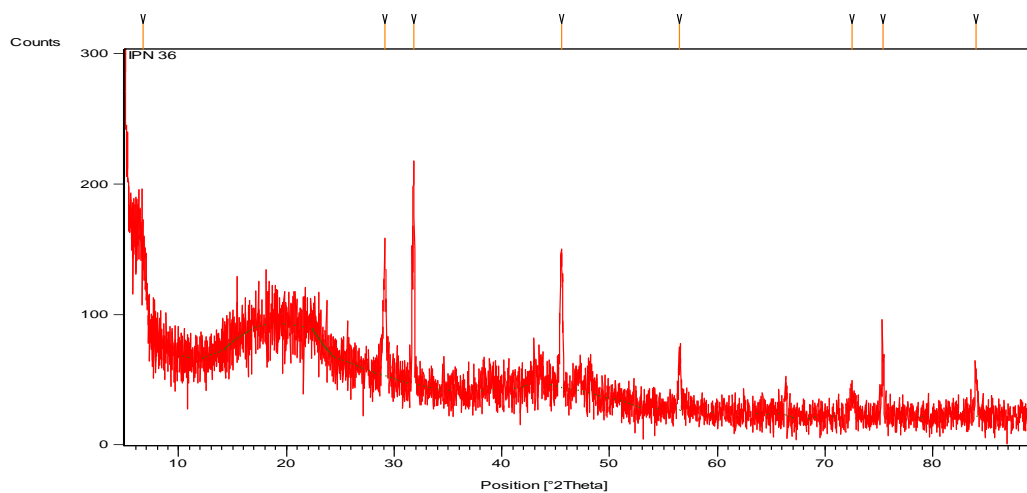


Fig 4d –WAXRD OF IPN 36

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
6.7354	82.44	0.9600	13.11282	51.00
29.1594	86.73	0.1920	3.06007	53.66
31.8412	161.64	0.2880	2.80819	100.00
45.5602	98.92	0.3360	1.98943	61.20
56.4868	39.27	0.2880	1.62779	24.29
72.5015	14.90	0.5760	1.30268	9.22
75.4015	35.32	0.2880	1.25961	21.85
84.0270	29.05	0.2880	1.15089	17.98

Table 4d

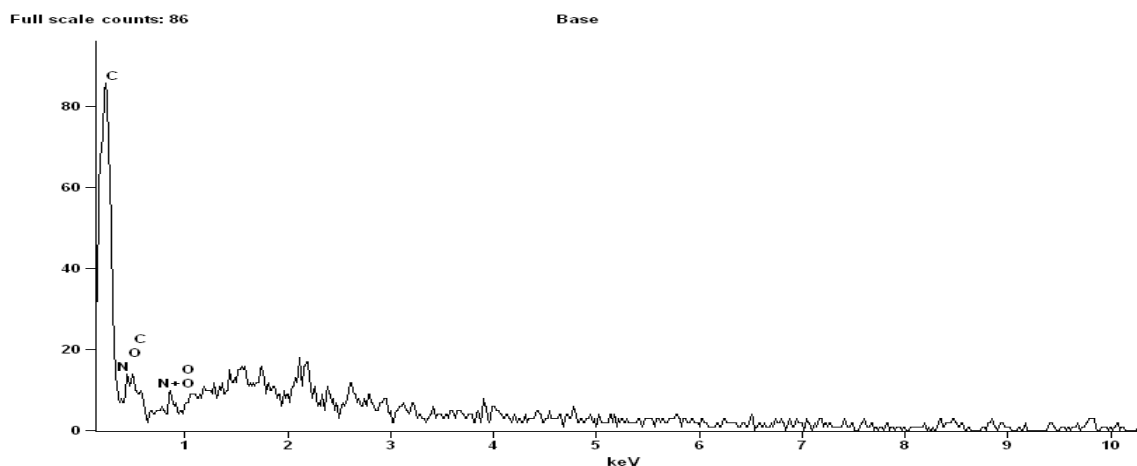


Fig 5a –IPN 27

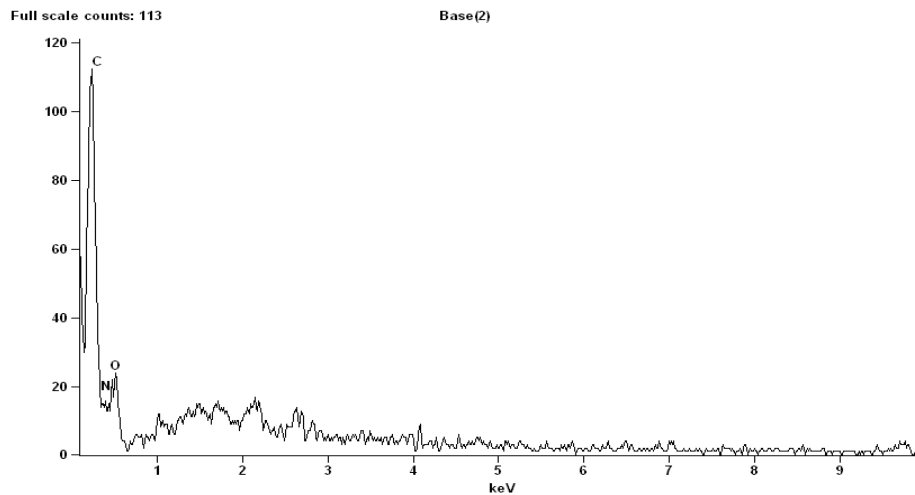


Fig 5b - IPN 32

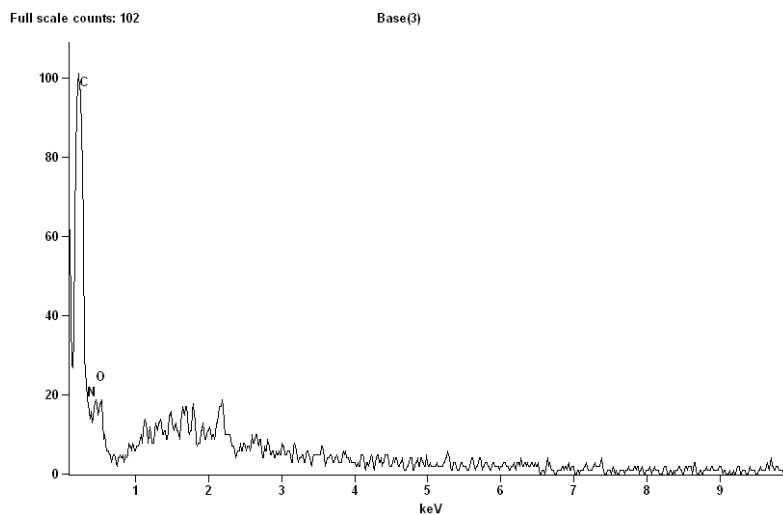


Fig 5c - IPN 35

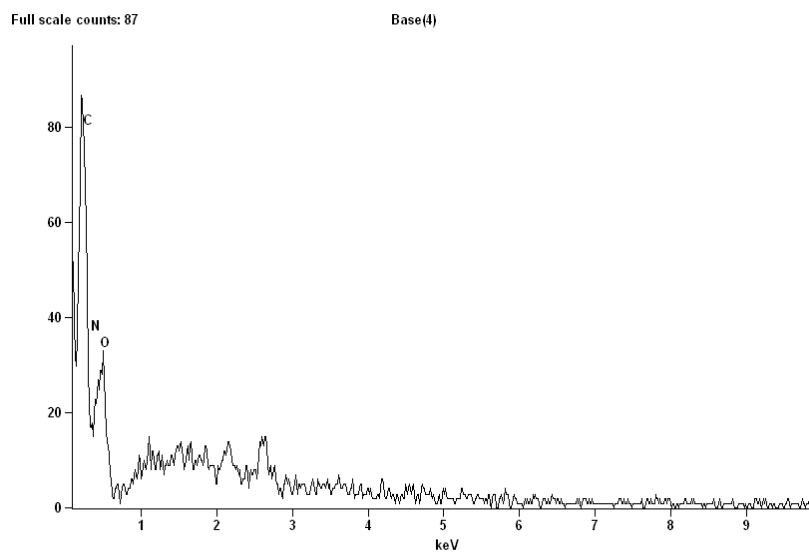


Fig 5d - IPN 36

SEM STUDY OF IPN 27

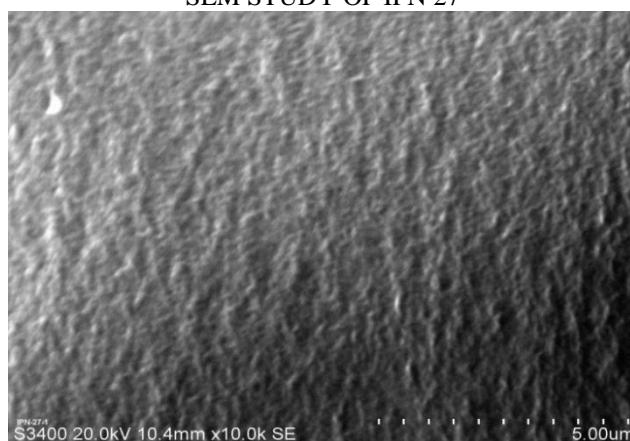


Fig 6a Fig 6b

SEM STUDY OF IPN 32

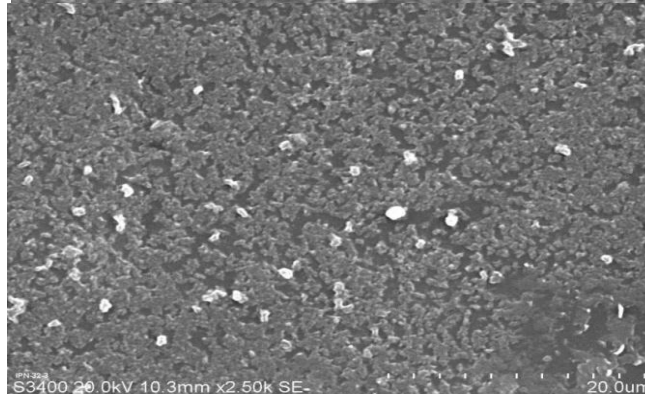
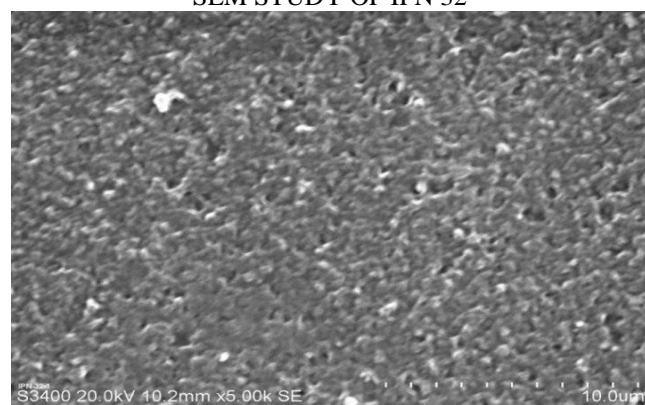


Fig 6c Fig 6d

SEM STUDY OF IPN 35

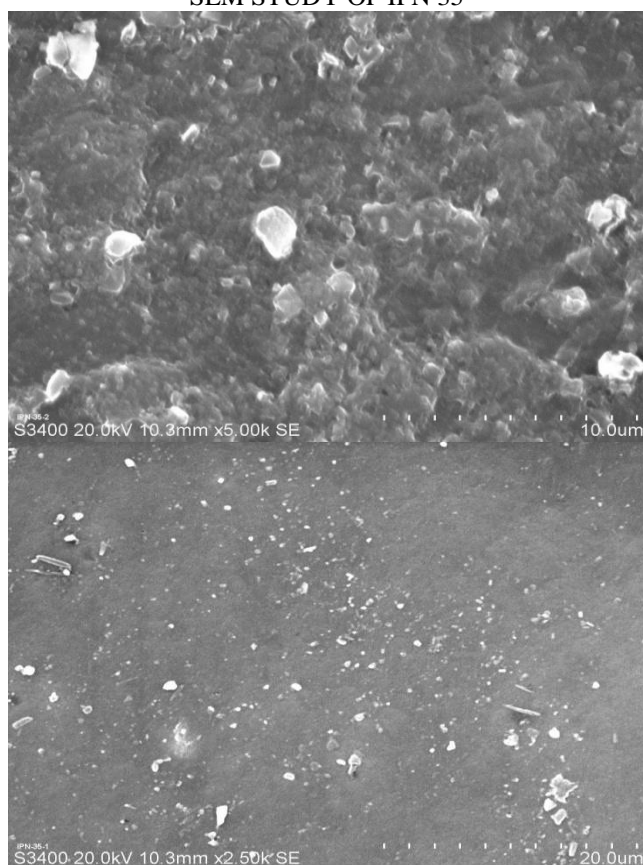


Fig 6e Fig 6f
SEM STUDY OF IPN 36

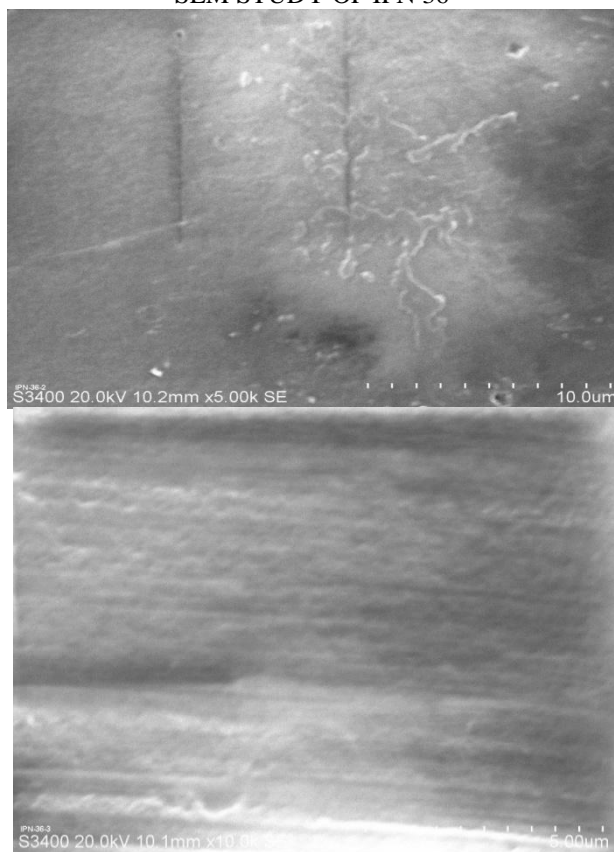


Fig 6g Fig 6h

Table 1. FEED COMPOSITION DATA

Sl. No.	Sample code	Composition	NCO/OH	PU/CBD
1	IPN 27	PS+DPMDI+CD of 3-amino benzoic acid	2.0	0.50/0.50
2	IPN 32	PS+DPMDI+CD of 4-amino benzoic acid	1.6	0.35/0.65
3	IPN 35	PS+DPMDI+CD of 4-amino benzoic acid	2.0	0.35/0.65
4	IPN 36	PS+DPMDI of 4-amino benzoic acid	2.0	0.50/0.50

Table-2 DSC PARAMETERS

Sl No.	Sample code	Composition of IPNs		PU/CBD Weight ratio	NCO/OH	Tg in °C	Tc in °C	Tm in °C
		PU	CBD					
1	IPN ₂₇	GSO+DPMDI	3-amino Benzoic acid	50:50	2.0	175.5	307.02	472.3
2	IPN ₃₂	GSO+DPMDI	4-amino Benzoic acid	35:65	1.6	137.2	420.1	420.6
3	IPN ₃₅	GSO+DPMDI	4-amino Benzoic acid	35:65	2.0	177.2	294.8 416.2	461.65
4	IPN ₃₆	GSO+DPMDI	4-amino Benzoic acid	50:50	2.0	162.5	294.82 416.6	461.7

Tg=glass transition temperature, Tc= curieing temperature, Tm= Melting point

Table-3a Percentage Of Ipn Samples Left After Thermal Degradation

Sl No.	Sample	Composition	Range of temperature						
			100	200	300	400	500	600	700
1	IPN ₂₇	MEP of SO + DPMDI + 3 amino Benzoic acid	98.4	93	76.4	57.6	21.9	17.8	17.8
2	IPN ₃₂	MEP of SO + DPMDI + 3 amino Benzoic acid	94.8	82.6	60.7	39.4	1.3	-	-
3	IPN ₃₅	MEP of SO + DPMDI + 3 amino Benzoic acid	99.3	94.6	82.5	67.8	22.55	19.9	19.2
4	IPN ₃₆	MEP of SO + DPMDI + 3 amino Benzoic acid	98.7	94.4	81.8	65.1	16.3	13.6	12.9

Table - 3b Kinetic parameters of different IPNs

Sample Codes	Temperature Range	Slope or Order of reaction	Activation Energy	Intercept
IPN27	140-300	0.2134	191.149	-0.1
	320-660	-1.8942	-691.19478	0.3616
IPN32	180-300	-3.996	-43.581972	0.0228
	300-440	-1.0014	-34.789118	0.0182
IPN 35	180-300	-10.395	6.116768	-0.0032
	320-580	-2.7442	-803.78155	0.4205
IPN 36	180-300	-10.704	26.378562	-0.0138
	320-580	-1.7569	-636.90847	0.3332

Table-4 PERCENTAGE OF DIFFERENT ELEMENTS IN THE IPNS QUANTITATIVE RESULTS BASE

CK	549	+/- 40	81.82	85.70	C
NK	0	+/- 11	0.00	0.00	N
OK	74	+/- 15	18.18	14.30	O
Total			100.00	100.00	

BASE 1 TABLE-4a

CK	681	+/- 45	77.61	82.19	C
NK	0	+/- 14	0.00	0.00	N
OK	133	+/- 16	22.39	17.81	O
Total			100.00	100.00	

BASE 2 TABLE-4b

CK	649	+/- 45	81.52	85.46	C
NK	0	+/- 15	0.00	0.00	N
OK	91	+/- 17	18.48	14.54	O
Total			100.00	100.00	

BASE-3 TABLE-4c

Comparison of Thermal Degradation, WAXRD Studies and SEM of Renewable Resources Based

CK	469	+/- 58	55.45	61.29	C
NK	27	+/- 40	14.77	14.00	N
OK	201	+/- 38	29.77	24.71	O
Total			100.00	100.00	

BASE 4 TABLE-4d

CK	442	+/- 54	56.50	61.91	C
NK	32	+/- 39	19.81	18.61	N
OK	127	+/- 25	23.69	19.48	O
Total			100.00	100.00	

BASE 5 TABLE-4e

CK	398	+/- 48	54.75	60.63	C
NK	24	+/- 36	14.91	14.16	N
OK	179	+/- 24	30.33	25.22	O
Total			100.00	100.00	

BASE 7 TABLE-4f

CK	375	+/- 45	60.22	65.69	C
NK	18	+/- 34	14.90	13.94	N
OK	110	+/- 21	24.88	20.37	O
Total			100.00	100.00	

BASE 8 TABLE-4g

CK	288	+/- 37	69.53	75.25	C
NK	0	+/- 11	0.00	0.00	N
OK	105	+/- 14	30.47	24.75	O
Total			100.00	100.00	

BASE 14 TABLE-4h

CK	344	+/- 45	69.60	75.02	C
NK	3	+/- 34	3.36	3.10	N
OK	101	+/- 21	27.04	21.88	O
Total			100.00	100.00	

BASE 15 TABLE-4i

CK	405	+/- 44	56.69	62.22	C
NK	27	+/- 34	17.83	16.78	N
OK	131	+/- 21	25.48	21.00	O
Total			100.00	100.00	

BASE 17 TABLE-4j

CK	229	+/- 26	80.63	84.72	C
NK	0	0	0.00	0.00	N
OK	35	35	19.37	15.28	O
Total			100.00	100.00	

BASE 19 TABLE-4k

Table-5 WAXRD STUDY

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
6.6666	90.67	0.7680	13.24802	19.91
27.5257	37.27	0.2880	3.23786	8.18
28.6765	56.19	0.2880	3.11048	12.34
31.9249	455.32	0.2400	2.80101	100.00
43.7147	23.36	1.1520	2.06904	5.13
45.7059	198.80	0.1440	1.98343	43.66
50.8830	16.76	1.1520	1.79311	3.68
56.7027	80.56	0.1920	1.62210	17.69
66.4155	41.35	0.2880	1.40648	9.08
72.5588	64.51	0.2880	1.30179	14.17
75.4177	52.92	0.2880	1.25938	11.62
84.1301	32.61	0.2880	1.14974	7.16

Pos. [$^{\circ}$ 2Th.]	Height [cts]	FWHM [$^{\circ}$ 2Th.]	d-spacing [\AA]	Rel. Int. [%]
6.7354	82.44	0.9600	13.11282	51.00
29.1594	86.73	0.1920	3.06007	53.66
31.8412	161.64	0.2880	2.80819	100.00
45.5602	98.92	0.3360	1.98943	61.20
56.4868	39.27	0.2880	1.62779	24.29
72.5015	14.90	0.5760	1.30268	9.22
75.4015	35.32	0.2880	1.25961	21.85
84.0270	29.05	0.2880	1.15089	17.98

TABLE-6 Decomposition By Bacteria And Virus

Sl. No	Sample Code	Initial mass	Mass after 15 days	Mass after 30 days	Mass after 45 days	Mass after 60 days
1	IPN27	10.42	10.36	10.23	10.11	10.01
2	IPN32	9.97	9.86	9.57	9.43	8.9
3	IPN35	9.62	9.42	9.4	9.32	9.29
4	IPN36	10.31	10.23	10.2	9.9	9.8