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 pallete 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 - Kα 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 0-5°C, 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 250°C in an inert nitrogen atmosphere taken in three naked flask fitted with a thermometer reflux condensers and a stirrer. At this temperature litherage (0.168g) and glycol(80ml) were added to the reaction mixture with constant stirring. The temperature was maintained at 210°C 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 75°C 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 75°C 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 75°C 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.

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**Scheme-1**

Triglyceride of linoleic (53%), oleic acid (18%) and linoleic acid (15%)

[Triglyceride of ricinoleic acid(87.5%),oleic(5%),linoleic(4%),linoleinic(0.5%)]

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Scheme 2

![Chemical structure](attachment:Scheme2.png)

4-amino benzoic acid

[Diazonium salt]

(i) $\text{C}_6\text{H}_4\text{OH} + \text{NaOH} \rightarrow \text{C}_6\text{H}_4\text{O}^-\text{Na}^+$

(ii) $\text{C}_6\text{H}_4\text{O}^-\text{Na}^+ + \text{H}_2\text{O} \rightarrow \text{C}_6\text{H}_4\text{OH} + \text{Na}^+$

(iii) $\text{C}_6\text{H}_4\text{OH} + \text{C}_6\text{H}_4\text{O}^-\text{Na}^+ \rightarrow \text{C}_6\text{H}_4\text{O}^-\text{C}_6\text{H}_4\text{O}^-\text{Na}^+$

Coupling

Cardanol based Dye (CD) with 4-amino benzoic acid

Scheme 3

![Chemical structure](attachment:Scheme3.png)

Polyol Modified Soyabeen Oil (PS)

diphenyl methane-4,4'-diisocyanate (DIIS)

450°C, 1 h Stirring

Scheme 4

(POLYURATHANE (PU))

![Chemical structure](attachment:Scheme4.png)

(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 3585.6 cm⁻¹, N-H stretching of >NH group at 3444.5 cm⁻¹, C-H stretching (ss/as) of >CH₂ and >CH₃ groups at 2871.9 cm⁻¹ and 2774.7 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 ofazo 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 ofazo 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 stretching 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 3535.4 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 2857.7 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 1407.4 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.

DSc. (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, Tg 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 = (Ts - Tr): Change in temperature

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\[ \Delta T \text{ is zero for no reaction} \]

Heat of fusion \( Q/m \)

\[ C_p = \frac{d}{dt} \times \frac{1}{m} \]

Percentage of crystallinity \( = \frac{\Delta T m - \Delta T c}{\Delta T m} \times 100 \)

**Conclusion**

From the table-2 it is evident that IPN\(_{27}\), IPN\(_{35}\), IPN\(_{36}\) are more thermally stable than IPN\(_{32}\) which has Tm value 420.6 and lowest molar ratio. While other three IPNs are comparatively more thermally stable.IPN\(_{32}\) has more Tg value than IPN\(_{27}\), IPN\(_{35}\), IPN\(_{36}\) 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:**

IPN\(_{32}\) is less stable at higher temperature than IPN\(_{27}\), IPN\(_{35}\), IPN\(_{36}\). All the three IPNs are stable upto 700°C polymer chain breaks nearly about 400°C to 450°C.

IPN\(_{27}\), IPN\(_{35}\), IPN\(_{36}\) all have same NCO/OH molar ratio (i.e. 2.0) are more stable than IPN\(_{32}\) 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.

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

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

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

**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 \( 2\Theta \) angle \( (2\Theta > 50) \) as shown in fig-4a, 4b, 4c, 4d and table-5a, 5b, 5c, 5d respectively.

**Conclusion**

From the above fact it is clear that IPN\(_{27}\) is more crystalline than IPN\(_{32}\), IPN\(_{35}\), IPN\(_{36}\) 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.

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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
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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
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Fig 3c – Freeman-Anderson Plot of IPN32

\[ y = -3.996x + 0.0228 \]
\[ R^2 = 0.8438 \]

Fig 3d – Freeman-Anderson Plot of IPN32

\[ y = -1.0014x + 0.0182 \]
\[ R^2 = 0.6356 \]

Fig 3e – Freeman-Anderson Plot of IPN35

\[ y = -10.39x - 0.003 \]
\[ R^2 = 0.760 \]

Fig 3f – Freeman-Anderson Plot of IPN35

\[ y = -2.744x - 0.420 \]
\[ R^2 = 0.327 \]
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Fig 3g – Freeman-Anderson Plot of IPN36

\[ y = -10.704x - 0.0138 \]
\[ R^2 = 0.755 \]

Fig 3h – Freeman-Anderson Plot of IPN 36

\[ y = -1.7569x + 0.3332 \]
\[ R^2 = 0.2242 \]

Fig 4a – WAXRD OF IPN 27
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Table 4a

Fig 4b – WAXRD of IPN 32

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Table 4b

Fig 4c – WAXRD of IPN 35

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Table 4c

Fig 4d – WAXRD OF IPN 36

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Table 4d

Fig 5a – IPN 27

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Fig 5b - IPN 32

Fig 5c - IPN 35

Fig 5d - IPN 36
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SEM STUDY OF IPN 27

Fig 6a  Fig 6b

SEM STUDY OF IPN 32

Fig 6c  Fig 6d
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SEM STUDY OF IPN 35

Fig 6e  Fig 6f

SEM STUDY OF IPN 36

Fig 6g  Fig 6h
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<th>Sl. No.</th>
<th>Sample code</th>
<th>Composition</th>
<th>NCO/OH</th>
<th>PU/CBD</th>
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<td>IPN 27</td>
<td>PS+DPMDI+CD of 3-amino benzoic acid</td>
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<td>0.50/0.50</td>
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<td>PS+DPMDI+CD of 4-amino benzoic acid</td>
<td>1.6</td>
<td>0.35/0.65</td>
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<td>2.0</td>
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<td>PS+DPMDI of 4-amino benzoic acid</td>
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Table 2 DSC PARAMETERS

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<th>PU/CBD Weight ratio</th>
<th>NCO/OH</th>
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<th>Tc in °C</th>
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<td>4</td>
<td>IPN 36</td>
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<td>2.0</td>
<td>162.5</td>
<td>294.8</td>
<td>461.7</td>
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Tg=glass transition temperature, Tc= curing temperature, Tm= Melting point

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

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Table - 3b Kinetic parameters of different IPNs

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

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

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BASE 19 TABLE-4k

**Table-5 WAXRD STUDY**

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**Decomposition By Bacteria And Virus**

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