# Polymers from Renewable Resources. I. Castor Oil-Based Interpenetrating Polymer Networks: Thermal and Mechanical Properties

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## SYNOPSIS

A number of polyurethanes (PUs) were synthesized by reacting castor oil with toluene-2,4-diisocyanate and hexamethylene diisocyanate and varying the NCO/OH ratio. All these polyurethanes were reacted with some acrylic monomers like ethyl acrylate, n-butyl acrylate, ethyl methacrylate, and butyl methacrylate using a crosslinker ethylene glycol dimethylacrylate and benzoyl peroxide as the initiator. The physicochemical properties of interpenetrating polymer networks (IPNs) are reported. Thermogravimetric analysis was used to study the thermal behavior of IPNs and the evaluation of kinetic parameters. Degradation mechanism has also been investigated. Some of the mechanical properties viz. tensile strength, shore-A hardness, elongation at break, etc. are also reported. © 1993 John Wiley & Sons, Inc.

## INTRODUCTION

The downstream plastic conversion industries have received great boost and have mushroomed without corresponding increase in the production of plastic raw materials in the world. There is a great shortage of plastic raw materials in the world,[1] especially in developing countries like India, Pakistan, and China. Recently, the use of renewable resources has attracted the attention of many researchers[2-8] because of their potential to substitute petrochemical derivatives. With petroleum production facing exhaustion day by day, chemists should focus their attention on renewable resources so that the polymers that will be required in the 21st century can be manufactured to feed the various industries.

The synthesis and characterization of interpenetrating polymer networks (IPNs) from natural products has attracted the attention of many research workers in the world.[9-25] Materials known as IPNs contain two or more polymers, each in network form. A practical restriction requires that at least one of the polymer networks has been formed (i.e.,
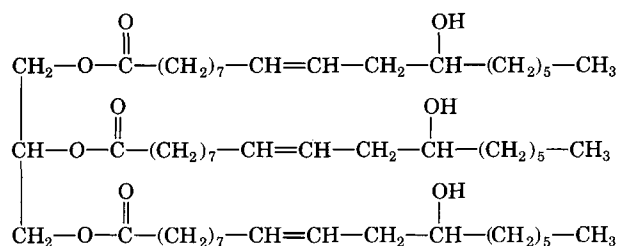
polymerized or crosslinked) in the immediate presence of the other. Two major types of IPNs synthesis have been explored.[26-45] The first, termed sequential IPNs, involves the preparation of a crosslinked polymer I, a subsequent swelling in network II monomer components, and polymerization of monomer II *in situ.*

The second type of synthesis yields materials known as simultaneous interpenetrating networks (SINs) and involves the mixing of all components in an early stage, followed by the formation of both networks via independent reactions proceeding in the same container. One network can be formed by a chain growth mechanism and the other by a step growth mechanism, which keeps the possibilities of grafting between the homopolymer networks at a minimum. Thus, for example, epoxy/acrylates,[21] polyurethane (PU)/polystyrene,[22] PU/polyacrylate,[23] and polyester/polystyrene[24] SIN systems have been synthesized.

This laboratory has taken up a research program for exploring the possibility of synthesizing a large number of industrially useful polymers from the forest products of Orissa, one of the states in India crowned with various forest products of oil-bearing wild plants like castor, tung, vernonia, linseed, crambe, lunaria, mahua, cashew nut, sal-seed, etc.

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It is planned to utilize some of these oils for the synthesis of novel interpenetrating polymer networks. Among the most interesting wild plant oils are the special functional group triglyceride oils. Although some of the triglyceride oils such as linseed and tung have only double bond functionality, a few others developed such functional groups as hydroxyl and epoxy.[8] One of the most naturally occurring vegetable oils is castor oil,[46-52] which has the advantage of possessing relatively high purity and being abundantly available in the forests of Orissa. This oil is extracted from the beans of the plant *Ricinum cummunis*. It is mainly used in Orissa as a burning oil in place of kerosine by the people in locally made small earthen pots. The oil is composed of essentially a triglyceride of hydroxy acid, *ricinoleic* acid, whose structure is given below:

$$
\begin{array}{l}
\underset{\displaystyle |}{CH_2}-O-\overset{\displaystyle O}{\overset{\displaystyle \|}{C}}-(CH_2)_7-CH=CH-CH_2-\overset{\displaystyle OH}{\overset{\displaystyle |}{CH}}-(CH_2)_5-CH_3 \\
\underset{\displaystyle |}{CH}-O-\overset{\displaystyle O}{\overset{\displaystyle \|}{C}}-(CH_2)_7-CH=CH-CH_2-\overset{\displaystyle OH}{\overset{\displaystyle |}{CH}}-(CH_2)_5-CH_3 \\
CH_2-O-\overset{\displaystyle O}{\overset{\displaystyle \|}{C}}-(CH_2)_7-CH=CH-CH_2-\overset{\displaystyle OH}{\overset{\displaystyle |}{CH}}-(CH_2)_5-CH_3
\end{array}
$$

Hence, castor oil is unique in possessing both unsaturation and nonconjugated hydroxyl function. The trifunctional nature of castor oil contributes toughness to the structure and the long fatty acid chain imparts flexibility. Due to its unusual structure, this oil is very much versatile in its applications. Although this oil was known from primitive ages for its medicinal values, it plays an important role in the field of polymer chemistry. Acting as a polyfunctional alcohol, castor oil reacts with polyfunctional isocyanates to form PUs that range from rigid foam to elastomers.[47,53-55] The hydroxyl function of the oil can also be made an important additive to impart plasticization in many polymer blends.

Castor oil also undergoes many familiar organic reactions to form useful derivatives. Epoxidized, hydrogenated, and hydroxylated oils are among the many derivatives. Epoxy derivatives are used to stabilize poly(vinyl chloride) resins and hydroxylated oil yields quick-drying alkyds.[48] The hydrogenated derivatives are used as greases and vehicle paints. Sebacic acid, a product of alkali fusion of castor oil, is used in the manufacture of nylon.[52]

This communication presents the preparation of a large number of IPNs using castor oil, a number of diisocyanates, and a multitude of vinyl monomers and some initiators. The chemical, thermal, and mechanical properties of the IPNs so prepared have been reported. It has also been planned to graft a number of vinyl monomers to the PUs or polyesters prepared from these oils by employing a multitude of initiating systems developed in this laboratory over the years.[56-60]

## EXPERIMENTAL

### Materials

Castor oil (refined) was used without any purification. It was obtained from the local market. Its characteristic values such as hydroxyl number, acid number, and isocyanate equivalent were determined by standard procedures and are furnished in Table I.

The chemicals used in this investigation were of analytic grade. Benzoyl peroxide was recrystallized from chloroform and vinyl monomer was freed from inhibitor before use. The isocyanate content was determined by titration with standard $n$-butyl amine with accuracy.

### Polyurethane Synthesis

Castor oil (55 g, 0.555 mol) was reacted with toluene-2,4-diisocyanate (TDI) (17.8 g, 0.099 mol) to maintain the NCO/OH ratio at 1.6. The reaction was carried out at 45°C with continuous stirring for 2 h. The prepolymer was isolated as a viscous liquid. Following this procedure, other PUs with varying NCO/OH ratios were prepared and also differing with diisocyanates.

### Synthesis of IPNs

IPNs were synthesized by charging the PU in different proportions into a round-bottomed flask. To this, the mixture of ethyl acrylate, 1% ethylene glycol dimethacrylate (EGDM), and 0.5% benzoyl peroxide was added. The mixture was stirred at room

**Table I  Properties of Castor Oil**

| | |
|---|---|
| Acid number (mg KOH/g) | 1.48 |
| Acid value | 2.45 |
| Hydroxyl number (mg KOH/g) | 160–162 |
| Hydroxyl value (meq/g) | 2.80 |
| Isocyanate equivalent | 330 |
| Molecular weight (mean) | 930 |
| Water content (wt %) | 0.21 |
| Specific gravity at 28°C | 0.965 |

temperature for 15 min to form a homogenous solution. The temperature was then raised to 60°C to initiate ethyl acrylate polymerization. After stirring for 1 h, the solution was poured into the glass mold kept in a preheated oven maintained at 60°C. It was kept at this temperature for 24 h and at 120°C for 4 h. The film thus formed was cooled slowly and removed from the mold with different compositions of same vinyl monomer and PU. Nine IPNs were synthesized and with different monomers a large number of IPNs were also prepared.

## CHARACTERIZATION

IPNs were characterized according to the following standard methods.

| Property | ASTM-Specifications |
|---|---|
| Resistance to chemical reagents | ASTM D543 (1978) |
| Mechanical properties | ASTM D638 (1971) |
| Hardness | ASTM D785 (1965) |

The thermal behavior was assessed on a Du Pont 951 Thermal Analyzer at a heating rate of 10°C/min in air. Tensile properties were studied on a Universal Instron Testing Machine.

## RESULTS AND DISCUSSION

The prepolymers were synthesized using castor oil, hexamethylene diisocyanate (HMDI), TDI, and diphenyl methane-4,4'-diisocyanate varying the NCO/OH ratio. The PUs were characterized from their infrared spectral data. IPNs were synthesized by varying the composition of prepolyurethane and acrylic monomers such as methyl acrylate, ethyl acrylate, $n$-butyl acrylate, methyl methacrylate, ethyl methacrylate, and butyl methacrylate. All IPNs were synthesized as tough films from transfer molding. The colors of these IPNs are from golden yellow to yellow. The IPNs are almost insoluble and their densities are heavier than water. The densities of all IPNs were determined and are furnished in Table II with data on feed composition of individual IPNs. The percentage change in weight loss of some of the IPNs as $IPN_2$, $IPN_4$, $IPN_6$, $\cdots$ $IPN_{16}$ were determined in $H_2SO_4$, $Ch_3COOH$, $HCl$, $HNO_3$, $NaCl$, $NaOH$, etc., and the results are furnished in Table III. There is almost no significant change in the physical appearance of all IPNs in the solvents under investigation but considerable weight loss was noticed in methyl ethyl ketone (MEK), $CCl_4$, $NaOH$, and toluene. This indicates that IPNs are partly soluble in some of these solvents and probably are not properly crosslinked. The IPNs are less resistant to alkali but much more resistant to acids.

**Table II    Data on Feed Composition of Individual IPNs with Specific Gravity**

| Sample Code | Used Diisocyanate/ Castor Oil | NCO/OH | Used Vinyl Monomer | Content of Prepolyurethane (wt %) | Content of Vinyl Monomer (wt %) | Specific Gravity (g/ml) |
|---|---|---|---|---|---|---|
| $IPN_1$ | TDI | 1.8 | BM | 25 | 75 | 1.152 |
| $IPN_2$ | TDI | 1.8 | BM | 35 | 65 | 1.041 |
| $IPN_3$ | TDI | 1.8 | BM | 45 | 55 | 1.290 |
| $IPN_4$ | HMDI | 1.8 | BM | 25 | 75 | 1.261 |
| $IPN_5$ | HMDI | 1.8 | BM | 35 | 65 | 1.323 |
| $IPN_6$ | HMDI | 1.8 | BM | 45 | 55 | 1.030 |
| $IPN_7$ | TDI | 1.6 | EM | 25 | 75 | 1.413 |
| $IPN_8$ | TDI | 1.6 | EM | 35 | 65 | 1.059 |
| $IPN_9$ | TDI | 1.8 | EM | 35 | 65 | 1.239 |
| $IPN_{10}$ | TDI | 1.8 | EM | 45 | 55 | 1.125 |
| $IPN_{11}$ | TDI | 1.6 | EA | 45 | 55 | 1.337 |
| $IPN_{12}$ | TDI | 1.8 | EA | 25 | 75 | 1.176 |
| $IPN_{13}$ | TDI | 1.8 | EA | 45 | 55 | 1.232 |
| $IPN_{14}$ | TDI | 2.0 | EA | 35 | 65 | 1.501 |
| $IPN_{15}$ | TDI | 2.0 | $n$-BA | 45 | 55 | 1.183 |
| $IPN_{16}$ | HMDI | 1.6 | $n$-BA | 45 | 55 | 1.218 |

Contents of prepolyurethane > 45% is discarded due to experimental difficulties. TDI, toluene-2,4-diisocyanate; HMDI, hexamethylene diisocyanate; BM, butyl methacrylate; EM, ethyl methacrylate; $n$-BA, $n$-butyl acrylate; EA, ethyl acrylate.

**Table III  % Weight Loss on Treatment Into Different Chemical Reagents**

| Chemical Reagents | IPN$_2$ | IPN$_4$ | IPN$_6$ | IPN$_8$ | IPN$_{10}$ | IPN$_{12}$ | IPN$_{14}$ | IPN$_{16}$ |
|---|---|---|---|---|---|---|---|---|
| 25% H$_2$SO$_4$ | 1.098 | 1.100 | 1.105 | 1.105 | 1.107 | 1.071 | 1.102 | 1.104 |
| 25% CH$_3$COOH | 10.65 | 5.010 | 6.085 | 5.210 | 5.216 | 8.13 | 6.024 | 5.213 |
| 15% HCl | 1.785 | 0.550 | 0.760 | 1.630 | 1.612 | 1.315 | 0.665 | 1.546 |
| 5% HNO$_3$ | 2.905 | 0.630 | 0.605 | 1.672 | 1.633 | 2.416 | 0.648 | 1.603 |
| 40% NaCl | 0.780 | 0.115 | 0.135 | 0.832 | 0.912 | 0.775 | 0.152 | 0.721 |
| 10% NH$_4$OH | 0.065 | 0.075 | 0.082 | 0.091 | 0.083 | 0.052 | 0.096 | 0.079 |
| 5% H$_2$O$_2$ | 0.215 | 1.005 | 1.113 | 0.633 | 0.751 | 0.182 | 1.115 | 0.561 |
| 5% NaOH | 1.825 | 13.25 | 15.23 | 11.35 | 12.47 | 1.910 | 14.85 | 10.40 |
| MEK[a] | 63.75 | 49.20 | 50.91 | 53.12 | 54.09 | 62.15 | 50.37 | 52.91 |
| CCl$_4$[a] | 56.15 | 26.05 | 28.32 | 35.61 | 32.29 | 53.05 | 27.11 | 34.44 |
| Toluene[a] | 86.35 | 66.00 | 69.01 | 72.01 | 73.25 | 82.23 | 68.04 | 70.09 |
| Distilled water | 0.825 | — | — | — | — | 0.795 | — | 0.711 |

MEK, methyl ethyl ketone.

[a] In solvents like MEK, toluene, and CCl$_4$, IPNs become brittle, two phases are separated without dissolution and the film loses elasticity.

[b] IPNs swell in 5% NaOH.

## Thermal Analysis

The $T_g$ thermograms of eight typical IPNs are presented in Figures 1 and 2 to give a comparative picture regarding the thermal stability. The characteristic thermal data, oxidation indices (OI) are presented in Table IV with the weight loss at different temperatures and the decomposition ranges. The oxidation index values calculated are based upon the weight of the carbonaceous charr related by the empirical equation[61]

$$OI \times 100 = 17.5 + 0.4 \times CR \qquad (1)$$

Kinetic parameters have been evaluated using the Freeman–Anderson[62] and Broido[63] methods, which provide the overall kinetic data averaged out for the experimental temperature range but possess the
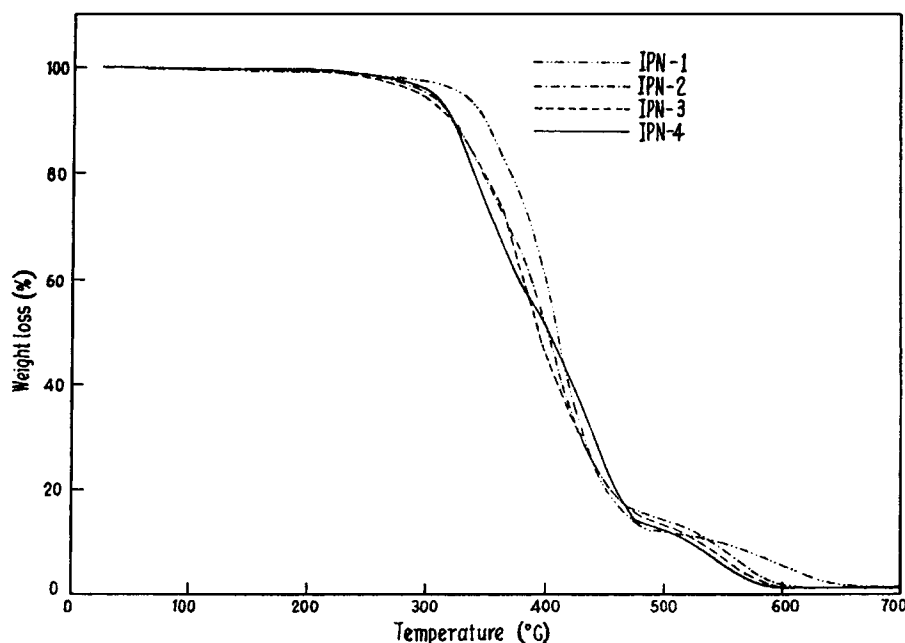


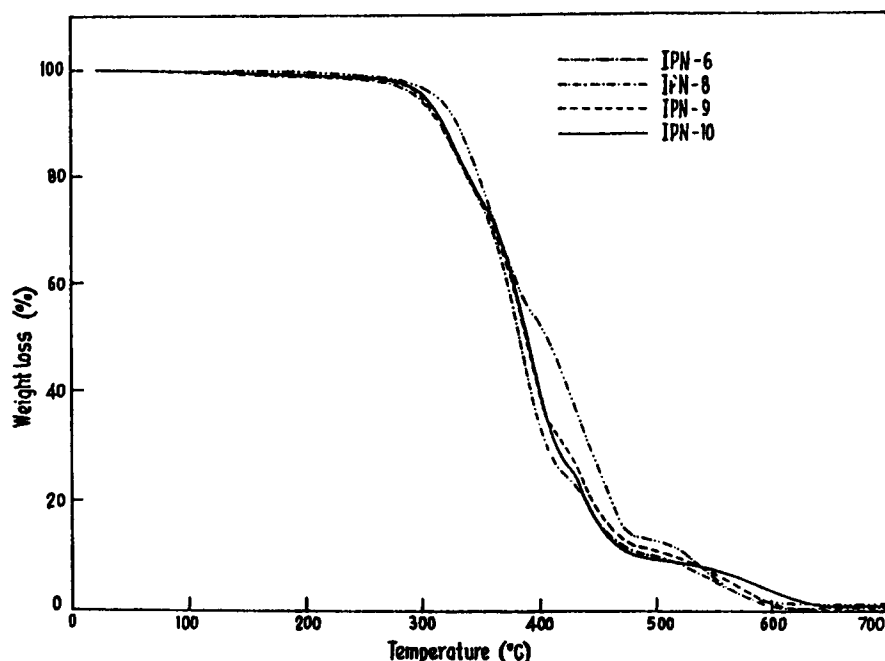**Figure 1**  $T_g$ thermogram of interpenetrating polymer networks.

**Figure 2**  $T_g$ thermogram of interpenetrating polymer networks.

added disadvantage that the observations at the extreme end are erratic and inexplicable. Here, for the sake of the calculations and to know the nature of decomposition, the complete thermogram is divided into three distinct sigmoids: as the IPNs under observation would not break down in a simple manner, for an individual sigmoid taking the residual weight of the pyrolyzed product at the beginning of that

sigmoid to be 100%, and as the change in morphological structure of the IPNs at each and every instant of pyrolysis do affect the rate of decomposition.

Although the above-cited methods involve a laborious method for the evaluation of kinetic data, they are attempted because of their merit for the single thermogram.[64,65] The Freeman–Anderson method makes use of the following equation, where

**Table IV  Characteristic Thermal Data**

| Sample Code | Decomposition Ranges | | | % wt Loss ($\pm 2\%$) up to Various Temperatures (°C) | | | OI[d] |
|---|---|---|---|---|---|---|---|
| | First[a] | Second[b] | Third[c] | 300 | 500 | 700 | |
| IPN$_1$ | 280–409 | 409–500 | 500–660 | 6.30 | 82.32 | 96.32 | 0.176 |
| IPN$_2$ | 251–403 | 403–460 | 460–600 | 7.15 | 84.67 | 98.99 | 0.175 |
| IPN$_3$ | 248–392 | 392–480 | 480–600 | 3.89 | 87.03 | 99.32 | 0.175 |
| IPN$_4$ | 247–401 | 401–470 | 470–590 | 4.76 | 90.65 | 99.21 | 0.176 |
| IPN$_5$ | 216–380 | 380–480 | 480–660 | 7.99 | 86.99 | 97.50 | 0.176 |
| IPN$_6$ | 264–409 | 409–470 | 470–600 | 6.68 | 58.12 | 98.77 | 0.175 |
| IPN$_7$ | 264–385 | 385–500 | 500–640 | 3.99 | 85.74 | 96.49 | 0.176 |
| IPN$_8$ | 250–385 | 385–480 | 480–620 | 5.12 | 86.32 | 98.52 | 0.175 |
| IPN$_9$ | 283–387 | 387–480 | 480–620 | 4.99 | 59.39 | 99.19 | 0.175 |
| IPN$_{10}$ | 275–389 | 390–500 | 500–640 | 5.01 | 92.22 | 96.58 | 0.175 |

Analyzed at a heating rate of 10°C/min in air.
[a] Weight loss due to retained moisture.
[b] Major decomposition stage.
[c] Secondary decomposition.
[d] $0.1 \times 100 = 17.5 + 0.4$ CR.

a plot $\Delta \log(-dw/dt)$ and $\Delta \log \bar{w}$ values (evaluated at a constant $\Delta 1/T$) gives the order of reaction $n$ as the slope and the energy of activation $E^*$ as the intercept:

$$\Delta \log(-dw/dt)$$

$$= n \, \Delta \log \bar{w} - (E^*/2.303R)(\Delta 1/T) \cdots \quad (2)$$

According to the Broido method

for $n = 1$, $\log \ln(1/y) = (E^*/2.303R)(1/T) + K$

$n = 2$, $\log(1/y - 1)$

$$= -(E^*/2.303R)(1/T) + K \cdots \quad (3)$$

where $k$ = any constant, $y$ = normalized weight ($W_t/W_0$), and $E^*$ = energy of activation obtained from the slope of the most appropriate plot. Freeman–Anderson and Broido plots of the IPNs prepared using TDI, ethyl methacrylate, butyl methacrylate, and HMDI are displayed in Figures 3 and 4, respectively, and the kinetic parameter data are tabulated in Table V.

A cursory glance at Table IV shows that all the IPNs under study decompose in three distinct temperature ranges. The initial slow weight loss around 250°C is attributed to moisture retained in the sample. The second weight loss (up to 60%) occurs between 200–400°C in all IPNs. This may be due to the decrosslinking of the IPNs under investigation. The third weight loss occurs during the range 500–
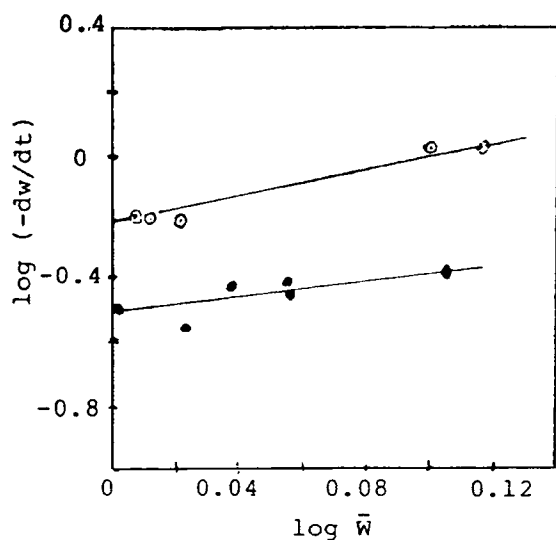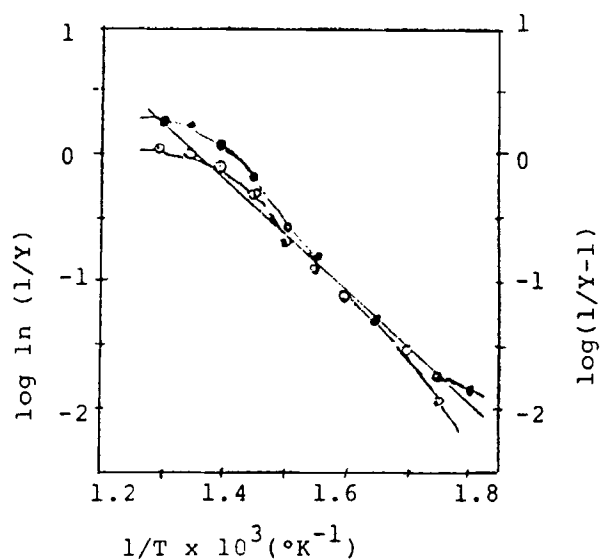


**Figure 4** Typical Broido plot for determination of activation energy ($E^*$) of IPN-8. (○) log ln $1/Y$ vs. $1/T$ × $10^3$; (●) log($1/Y - 1$) vs. $1/T$ × $10^3$.

700°C, indicating the complete decomposition of the IPNs.

A perusal of the thermograms indicate that all the IPNs decompose at a very high temperature, around 700°C. The IPNs prepared using TDI and butyl methacrylate decompose at higher temperature than those prepared using TDI and ethyl methacrylate. This is quite evident because the poly(butyl methacrylate) grafted to PU would be bulkier and more crosslinked in comparison to IPNs prepared from TID and ethyl methacrylate. Further, in the case of $IPN_1$ the content of monomer % is 75



**Figure 3** Typical Freeman–Anderson plot for determination of activation energy ($E^*$) and order of reaction ($n$). (●) IPN-1; (○) IPN-8.

**Table V  Kinetic Parameters**

| Sample Code | Energy of Activation (kcal/mol) $E^*$ | | Order of Reaction (A) ($n$) |
|---|---|---|---|
| | A | B | |
| $IPN_1$ | 27.63 | 26.32 | 2.6 |
| $IPN_2$ | 24.20 | 25.00 | 1.8 |
| $IPN_3$ | 17.95 | 18.12 | 1.6 |
| $IPN_4$ | 20.30 | 20.12 | 2.3 |
| $IPN_5$ | 23.10 | 22.90 | 1.5 |
| $IPN_6$ | 30.24 | 30.71 | 2.9 |
| $IPN_7$ | 19.20 | 20.10 | 1.3 |
| $IPN_8$ | 22.10 | 21.49 | 2.0 |
| $IPN_9$ | 18.51 | 17.80 | 1.9 |
| $IPN_{10}$ | 25.30 | 24.90 | 2.2 |

A, Freeman–Anderson method; B, Broido method.

whereas the content of monomer % in $IPN_2$ is 65 and that in $IPN_3$ is 55. $IPN_1$ decomposes at a higher temperature, i.e., at 660°C, compared to $IPN_2$ and $IPN_3$, which decompose around 600°C because of low monomer content. This is also evident from the well-known fact that, poly(butyl acrylate) is more stable than poly(ethyl acrylate). Comparing the IPNs prepared by TDI and ethyl methacrylate, it is observed that $IPN_7$, having 75% monomer weight to 25% PU weight, decomposes at a higher temperature than $IPN_8$, $IPN_9$, and $IPN_{10}$, where the percentage of monomer content is less.

Comparing the IPNs prepared using HMDI and TDI, it is seen that those prepared using TDI decompose at a higher temperature than those prepared using HMDI. This can be explained by considering the fact that the IPNs prepared from TDI possess the aromatic ring whereas those prepared from HMDI contain an open-chain hydrocarbon system.

Although specific analytic procedures were not followed but basing upon the mass of carbonaceous charr, it can be concluded that the IPNs are not good flame retardants, as evidenced by low oxygen index. Perusal of Table IV shows that the oxygen index values are almost same in all IPNs.

The values of energy of activation for decomposition and order of reaction were obtained using the Freeman–Anderson and Broido methods. The values of the activation energy lie between 18.50–30.20 kcal/mol using the Freeman–Anderson method and 17.80–30.70 kcal/mol using the Broido method. This indicates that activation energies calculated using both methods are almost the same, indicating accuracy of the calculation. The order of reaction lies in the range of 1–3, thereby indicating that the decomposition of the IPNs is complicated and occurs in three different steps, the first step being the loss of moisture absorbed, the second the decrosslinking of the IPNs, and the third the complete decomposition of the IPNs. Because the IPNs are complicated in structure, it is difficult to predict the way of decomposition with respect to the increase of temperature. Further work to predict the actual method of decomposition is under study.

## Mechanical Properties

Some mechanical properties such as elongation at break (%), tensile strength, and shore-A hardness are presented in Table VI. The properties of the IPNs are compared with those of homopoly(methyl acrylate) and poly(butyl acrylate). It is evident that the polyacrylates are harder and brittle in nature.

**Table VI    Data on Mechanical Propeties**

| Sample Code | Tensile Strength (psi) | % Elongation at Break | Shore-A Hardness |
|---|---|---|---|
| $IPN_{11}$ | 320.0 | 38.0 | 72.0 |
| $IPN_{12}$ | 185.0 | 110.0 | 68.0 |
| $IPN_{13}$ | 192.0 | 102.0 | 90.0 |
| $IPN_{14}$ | 145.0 | 250.0 | 55.0 |
| $IPN_{15}$ | 199.0 | 75.0 | 85.0 |
| $IPN_{16}$ | 215.0 | 162.0 | 80.0 |

Interpenetration of PU as a separate phase in polyacrylates brings about the enhanced modification in mechanical properties such as elongation at break (%), tensile strength, shore-A hardness, etc.

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