

Interpenetrating Polymer Networks of Poly(butyl methacrylate) and Poly(α -terpineol-co-styrene): Synthesis and Characterization

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Received 14 October 2004; accepted 20 May 2005

DOI 10.1002/app.23114

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Simultaneous interpenetrating polymer networks (IPNs) based on poly(butyl methacrylate) and poly(α -terpineol-co-styrene) were synthesized with azobisisobutyronitrile (AIBN) as the initiator and divinyl benzene as the crosslinking agent in xylene under an inert nitrogen atmosphere. Fourier transform infrared spectra provided structural evidence for the IPNs, indicating characteristic frequencies of ester groups of butyl methacrylate at 1723 cm^{-1} and alcoholic groups of α -terpineol at 3436 cm^{-1} . Scanning electron microscopy revealed threadlike network structures. Properties such as percentage swelling and average molecular weight between crosslinks were direct func-

tions of the copolymer and initiator (AIBN) concentrations and inverse functions of the monomer (butyl methacrylate) and crosslinking agent (divinyl benzene) concentrations. Differential scanning calorimetry showed an IPN glass-transition temperature at 80.2°C. The thermal decompositions of the IPNs were established with the help of thermogravimetric analysis. The value of the activation energy, calculated from thermogravimetric analysis with the Coats and Redfern equation, was 23 kJ/mol. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 343–352, 2006

Key words: crosslinking; morphology; networks; swelling

INTRODUCTION

Despite the large number of polymers that have been synthesized, the restless and probing minds of scientists, with respect to the need for ever-expanding technology, have continually sought to prepare high-performance polymers with unique and useful properties. Among polymers of recent interest, considerable attention has been given to the formation of interpenetrating polymer networks (IPNs). IPNs are defined as a combination of two polymers in network form, at least one of which is synthesized and/or crosslinked in the immediate presence of the other.^{1,2} IPNs have the following characteristics:^{3,4}

1. An IPN swells but does not dissolve in solvents.
2. IPNs show various degrees of phase separation in their morphologies.

IPNs can readily be modified to enhance specific capabilities and, hence, are promising materials for a variety of industrial applications in diverse fields, such as ion-exchange resins, reinforced elastomers,

thermoplastic IPNs, piezodialysis membranes, high-temperature alloys, coatings and adhesives, noise- and vibration-damping materials, and sheet-molding compounds (for transportation, appliances, and electrical applications).

The field of IPNs was laid down by Millar⁵ and continued by Sperling and coworkers,^{6,7} Frisch et al.,⁸ and Lipatov et al.,^{9,10} who presented articles on the synthesis, morphology, and mechanical behavior of IPNs. A detailed and reliable understanding has been developed on monomer functionality and polymer network formation.¹¹ Several general reviews on IPNs have emphasized the relationship between the synthesis and properties,^{12–14} self-organization,¹⁵ and industrial applications.¹⁶ Further, there is splendid scientific literature on IPNs based on polyurethane,¹⁷ polyethylene and poly(butyl methacrylate-co-styrene),¹⁸ polyacrylate and poly(styrene-co-methyl methacrylate) latex,¹⁹ polyethylene and poly(butyl methacrylate-co-methyl methacrylate),²⁰ poly(*n*-butyl acrylate) and polystyrene,²¹ and polybutadiene and poly(methyl methacrylate).²² The literature has also been enriched by several reports on the synthesis of IPNs with one metal acrylate, such as Zn; Cr with vinyl monomers;^{23,24} and two metal acrylates.²⁵ However, reports on the syntheses of IPNs containing terpenes are still scarce; for example, the *D*-borneol-poly(bismuth acrylate) IPN²⁶ and poly(citronellol-*alt*-methyl methacrylate) and polystyrene IPN²⁷ are of recent origin and were reported in

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Contract grant sponsor: DST (New Delhi, India); contract grant number: SP/S1/H-26/2000.

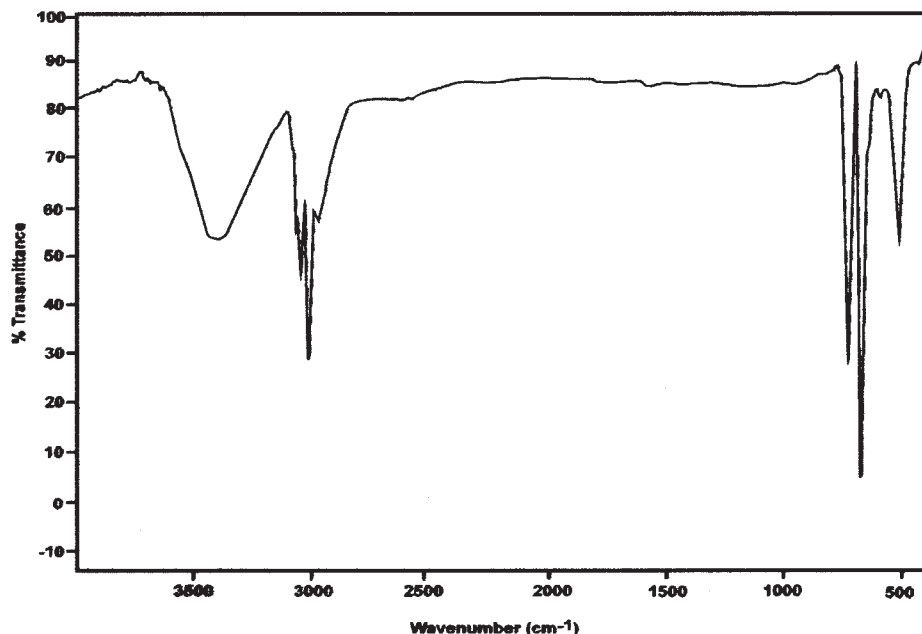


Figure 1 IR spectrum of the α -terpineol-co-styrene copolymer.

the literature for the first time by researchers in this laboratory.

Terpenes have played an important role in green chemistry, and their potential as novel monomers in the field of copolymerization have been recently established.²⁸⁻³⁰ α -Terpineol



a monocyclic monoterpene, is the oxygenated derivative of terpenes and possesses important flavor and perfume characteristics, having a hydroxy functional group. Recently, it was copolymerized with butyl methacrylate (BMA),³¹ and methyl methacrylate,³² but it has never been used in the synthesis of an IPN.

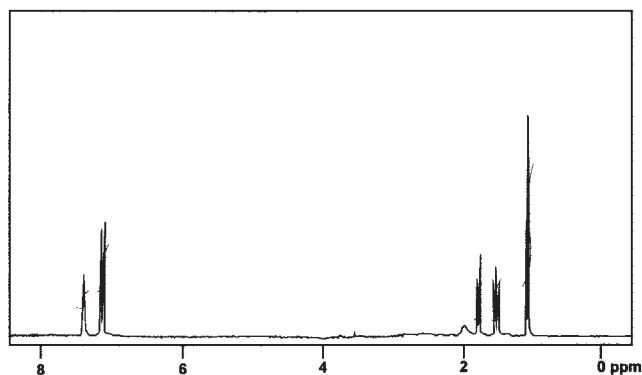


Figure 2 ^1H -NMR spectrum of the α -terpineol-co-styrene copolymer.

Therefore, this article highlights the synthesis and characterization of IPNs of α -terpineol-co-styrene with BMA.

EXPERIMENTAL

Styrene and BMA (Merck-Schuchardt) and other solvents were purified by the usual methods and distilled *in vacuo*.³³ Divinyl benzene (DVB; Merck) was used without further purification. Azobisisobutyronitrile (AIBN) was recrystallized with methanol (mp = 102°C). α -Terpineol (Merck) ($M = 154.25$ g/mol; 1 L = 0.94 kg; bp = 217°C; $n_D^{20} = 1.4813$) was used after fractional distillation.

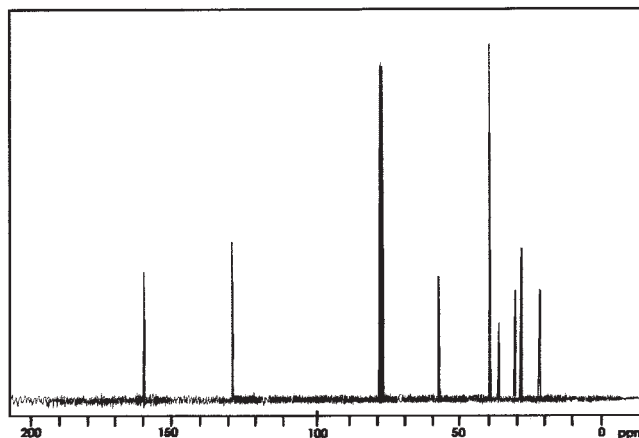
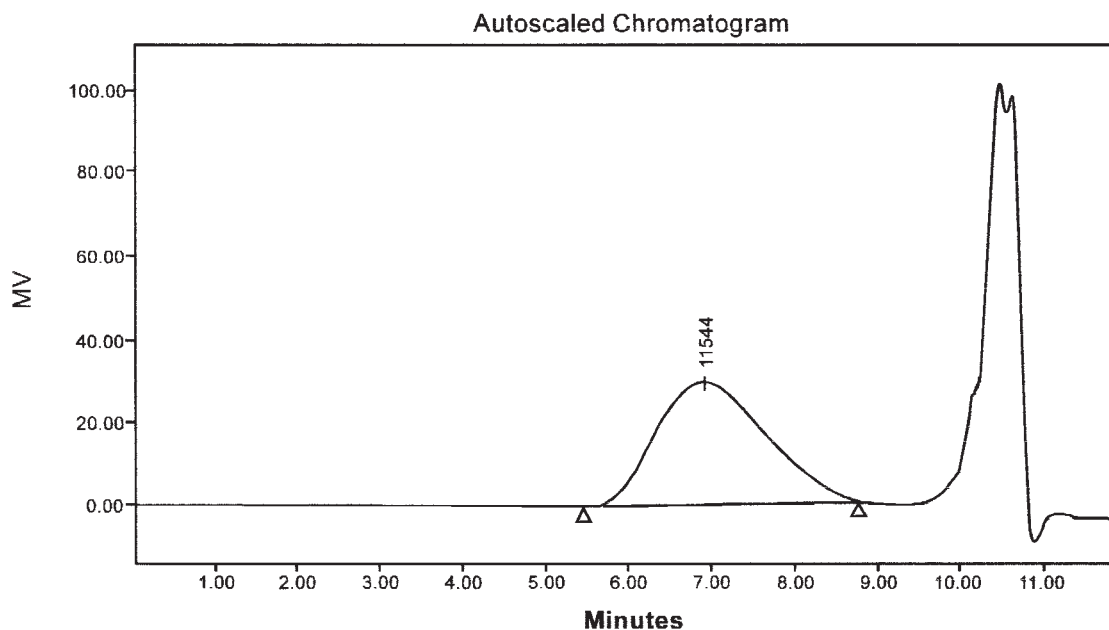


Figure 3 ^{13}C -NMR spectrum of the α -terpineol-co-styrene copolymer.



GPC Results

	Dist Name	Elution Volume (ml)	Retention Time (min)	Adjusted RT (min)	Mn	Mw	MP	Mz	M_{z+1}	Mz/Mw
1		6.893	6.893	6.893	7767	11338	11544	15028	18144	1.325451

GPC Results

	M_{z+1}/M_w	Area ($\mu\text{V}\cdot\text{sec}$)	%Area	Height (μV)	% Height	Integration Type	Peak Codes	Points Across Peak	Start Time (min)
1	1.600354	2810238	100.00	30056	100.00	BB		199	5.450

GPC Results

	End Time (min)	Baseline Start (min)	Baseline End (min)	Slope ($\mu\text{V}/\text{sec}$)	Offset (μV)
1	8.767	5.450	8.767	8.383192e-001	-2.724585e+000

Figure 4 GPC graph and results of the α -terpineol-co-styrene copolymer. M_n = number-average molecular weight; M_w = weight-average molecular weight; M_z = z-average molecular weight.

Characterization

IR, $^1\text{H-NMR}$, and $^{13}\text{C-NMR}$ spectra of the copolymer were recorded with a PerkinElmer 599B (with KBr pellets) and Varian 100 HA and Jeol 400 LA spectrophotometers, respectively, with CDCl_3 as the solvent and tetramethylsilane as the internal reference. Gel permeation chromatography (GPC) studies of the copolymer were made with a Shimadzu gel permeation chromatograph (model CR 7A). The elution solvent was tetrahydrofuran at a temperature of 25°C . The measurement of the glass-transition temperature (T_g)

of the copolymer and the IPNs was carried out with a PerkinElmer Pyris Diamond differential scanning calorimeter at a heating rate of $10^\circ\text{C}/\text{min}$ under a nitrogen atmosphere.

Thermogravimetric analysis (TGA) of the copolymer and IPNs were recorded by a PerkinElmer Pyris Diamond thermogravimetric analyzer at a heating rate of $10^\circ\text{C}/\text{min}$ under a nitrogen atmosphere.

Differential scanning calorimetry (DSC) of poly(butyl methacrylate) was carried out on a MDSC 2910 modulated differential scanning calorimeter (TA In-

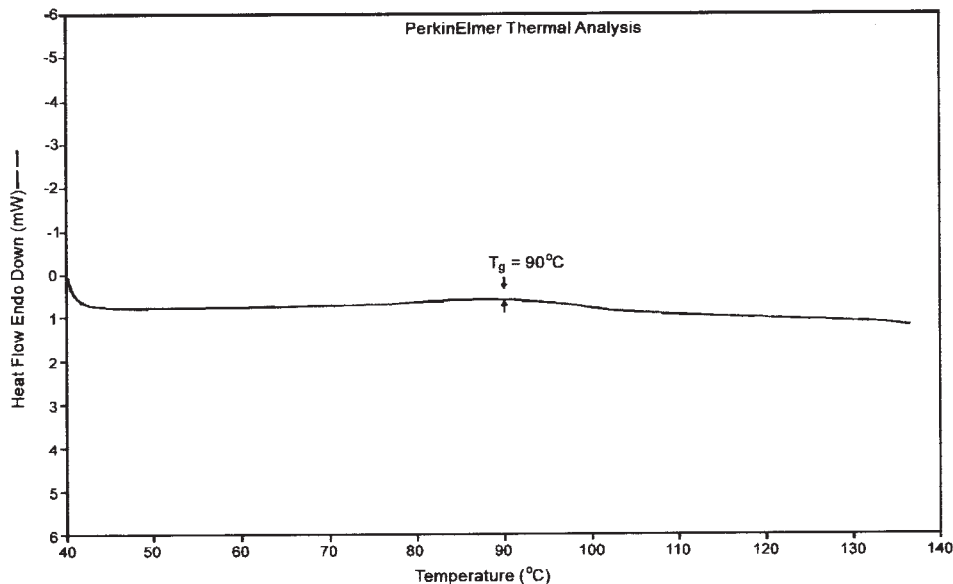


Figure 5 DSC curve of the α -terpineol-co-styrene copolymer heated from 40 to 140°C at 10°C/min.

struments) at a heating rate of 10°C/min under a nitrogen atmosphere. TGA of poly(butyl methacrylate) was measured with a Hi-Res TGA 2950 thermogravimetric analyzer (TA Instruments) attached to a thermal analyst 2100 (DuPont Instruments) thermal analyzer at a heating rate of 10°C/min under a nitrogen atmosphere.

Scanning electron microscopy (SEM) morphology

The morphology of the IPNs were studied by SEM with a resolution of 50 Å and a depth of field 30 μ m. Samples were mounted on a SEM stub with a graphite

adhesive paste and coated with gold in a SEM coating unit. The samples were then scanned in a Jeol JSM 840A scanning electron microscope.

Synthesis of the copolymer

The synthesis and characteristics of the copolymer of α -terpineol with styrene was reported elsewhere.³⁴ Briefly, a solution in xylene containing α -terpineol (0.730 mol/L), styrene (1.51 mol/L), and AIBN (7.6×10^{-3} mol/L) as the initiator was polymerized for 2 h at $80 \pm 0.1^\circ\text{C}$ under an inert atmosphere of nitrogen. The copolymer was precipitated in acidified

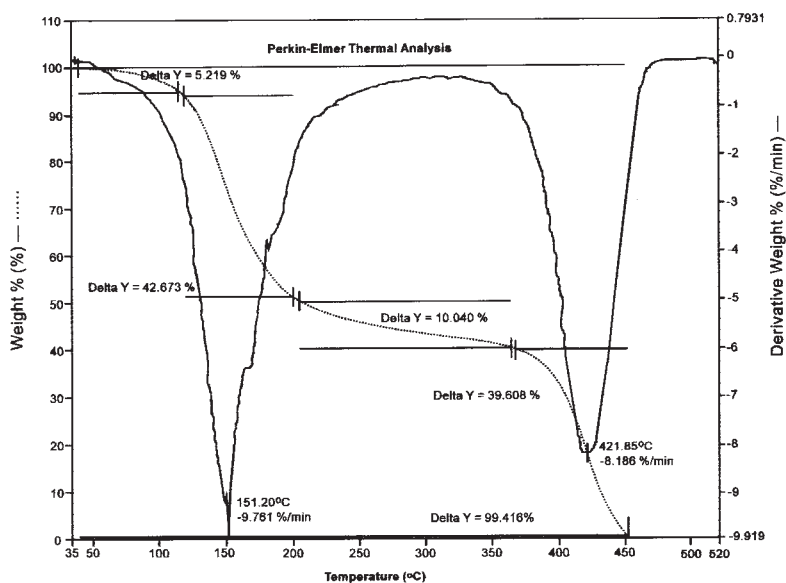


Figure 6 TGA curve of the α -terpineol-co-styrene copolymer heated from 35 to 530°C at 10°C/min.

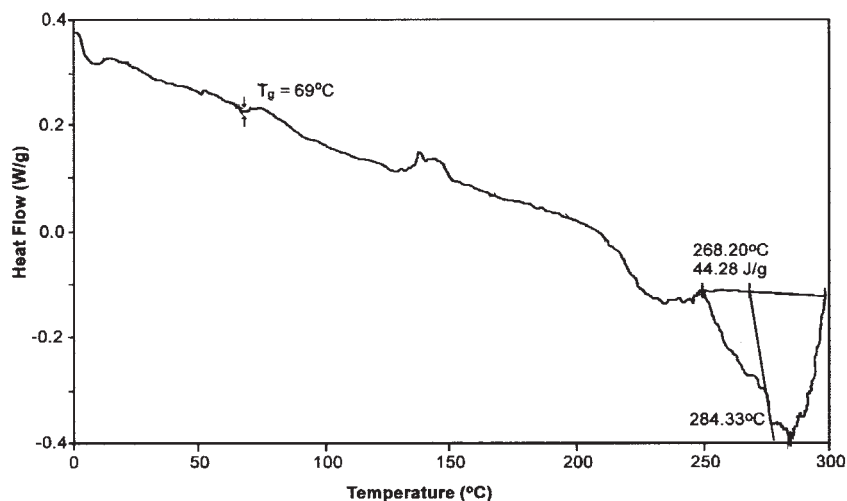
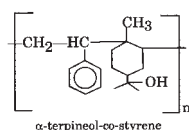


Figure 7 DSC curve of poly(butyl methacrylate).

methanol and dried to a constant weight. It was then refluxed with cyclohexane (to remove the homopolymer) and dried to a constant weight. The structure of the copolymer of α -terpineol with styrene was as follows:



Characterization of the α -terpineol-co-styrene copolymer³⁴

Ir spectroscopy³⁴

The IR spectrum of the copolymer (Fig. 1) showed the following group of bands:

1. The range of the C—H stretching band due to the aromatic ring of polystyrene was at 3025.93 cm^{-1} .
2. The range of the C—H bending vibration showed bands at 756.56–699.29 cm^{-1} .
3. The alcoholic band of α -terpineol was at 3409.09 cm^{-1} .

NMR spectroscopy

¹H-NMR³⁴. The ¹H-NMR spectrum (Fig. 2) of the copolymer exhibited the following peaks: a singlet (3H, CH₃) at 0.9 δ , a doublet (2H, CH₂) at 1.3–2.3 δ , a triplet (1H, CH) at 1.8–2.2 δ , a doublet (5H, C₆H₅) at 6.5–7.3 δ , and a singlet (1H, OH) at 7.4–7.8 δ . The large integral value for the —OH peak as compared with the five aromatic protons may have been due to the fact that the α -terpineol, apart from being polymerized, also underwent chain transfer to monomers from

polymers or the radical initiator. The ¹H-NMR spectra of the copolymer confirmed the formation of the functional copolymer.

¹³C-NMR³⁴. The ¹³C-NMR spectra (Fig. 3) of the copolymer displayed the following peaks: 13–22 δ for CH₃, 23–25 δ for CH₂, 55–80 δ for C—OH, and 120–140 δ for CH₂.

Copolymer composition and reactivity ratio values³⁴

The relative area of the peaks at 6.5–7.3 δ due to the phenyl protons of styrene and at 7.4–7.8 δ due to the hydroxy proton of the α -terpineol unit in the ¹H-NMR spectrum permitted us to estimate the copolymer composition. The reactivity ratio values [r_1 (styrene) = 0.033; r_2 (α -terpineol) = 0.004] were calculated by the Kelen–Tüdös method. The product $r_1 r_2$ was nearly zero, which was a sign of alternating copolymerization. Therefore, the possibility of the coexistence of graft, , and random copolymers was ruled out.

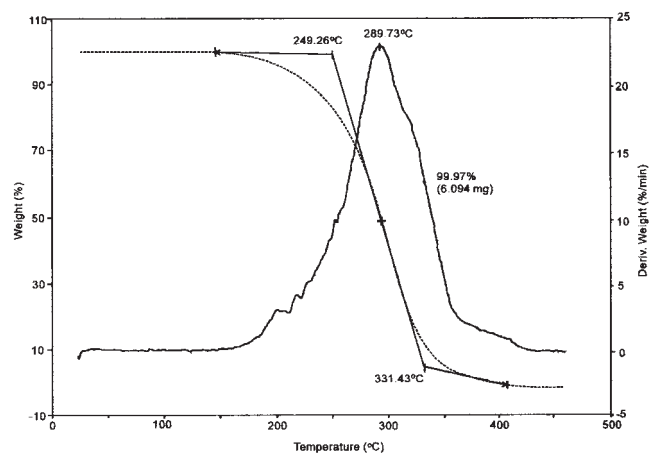


Figure 8 TGA curve of poly(butyl methacrylate).

TABLE I
Variable Manipulation in Simultaneous IPNs

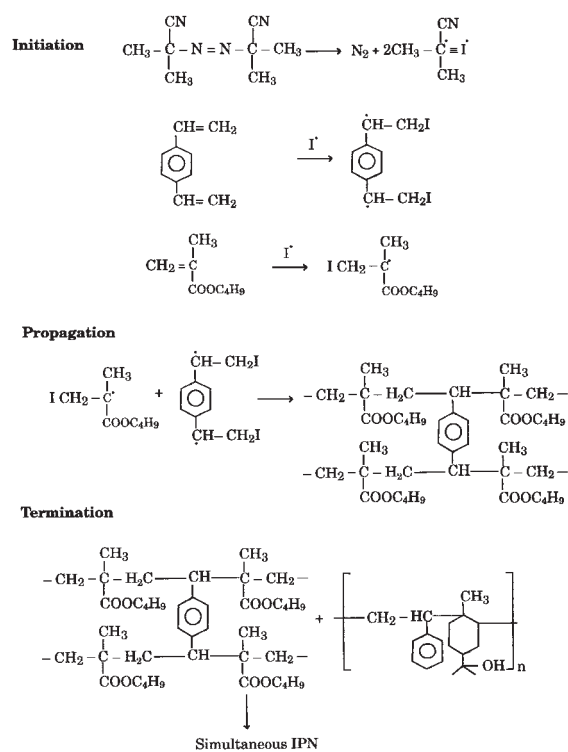
Sample	[Poly(α -terpineol- <i>co</i> -styrene) _(base M) × 10 ² (mol/L)]	[BMA] (mol/L)	[AIBN] × 10 ³ (mol/L)	[DVB] (mol/L)
IPN-1	0.05	0.89	5.23	0.76
IPN-2	1.10	0.89	5.23	0.76
IPN-3	1.66	0.89	5.23	0.76
IPN-4	2.21	0.89	5.23	0.76
IPN-5	3.32	0.89	5.23	0.76
IPN-6	1.66	0.36	5.23	0.76
IPN-7	1.66	1.25	5.23	0.76
IPN-8	1.66	1.79	5.23	0.76
IPN-9	1.66	2.15	5.23	0.76
IPN-10	1.66	0.89	1.74	0.76
IPN-11	1.66	0.89	8.71	0.76
IPN-12	1.66	0.89	10.45	0.76
IPN-13	1.66	0.89	5.23	0.25
IPN-14	1.66	0.89	5.23	0.51
IPN-15	1.66	0.89	5.23	1.02
IPN-16	1.66	0.89	5.23	1.56

GPC

A typical GPC curve and other data are shown in Figure 4.

Thermal analysis

DSC. The DSC scan of the copolymer (Fig. 5) showed the T_g as 90°C. The value was in agreement with



Scheme 1

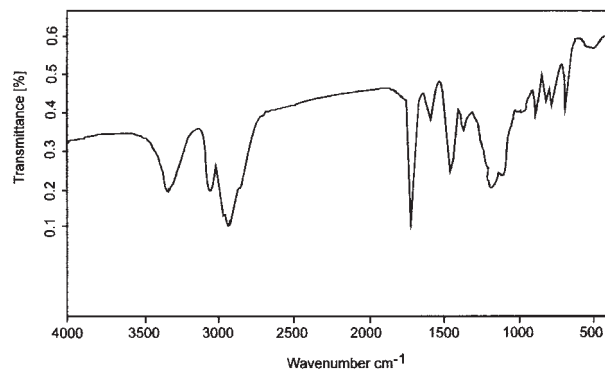


Figure 9 FTIR spectrum of IPN-3.

values reported in literature for other terpene copolymers with vinyl monomers (i.e., geraniol-*co*-styrene $T_g = 90^\circ\text{C}$,³⁵ citronellol-*co*-acrylonitrile $T_g = 102^\circ\text{C}$,³⁶ and linalool-*co*-vinyl acetate $T_g = 85^\circ\text{C}$ ³⁷).

TGA. The TGA curve for the copolymer (Fig. 6) exhibited weight loss with temperature.^{38,39} The copolymer was stable up to 140°C. The thermal behavior data were as follows:

1. The onset of major weight loss was around 150°C; the completion of major weight loss occurred at 421°C.
2. A total weight loss of 99.4% was observed in the range 35–450°C.

Characterization of poly(butyl methacrylate)

Thermal analysis

DSC. The DSC curve (Fig. 7) indicated that the T_g of poly(butyl methacrylate) was 69°C.

TABLE II
Percentage Swelling of IPN in Different Solvents and Percentage Extractable Material

Sample	Solvent					Extractable material (%)
	DMF	DMSO	Dioxane	Toluene	Benzene	
IPN-1	58	51	30	23	21	25
IPN-2	69	55	46	33	24	18
IPN-3	85	72	50	37	32	15
IPN-4	95	81	53	42	38	12
IPN-5	121	109	61	50	42	11
IPN-6	94	86	68	59	45	30
IPN-7	69	58	47	32	29	13
IPN-8	51	49	41	28	25	10.5
IPN-9	48	42	38	25	22	9.8
IPN-10	71	61	42	35	25	26
IPN-11	99	79	71	60	59	12.5
IPN-12	105	92	84	69	66	11.0
IPN-13	102	96	80	68	61	24.6
IPN-14	94	81	72	48	39	20
IPN-15	66	61	45	38	27	11.3
IPN-16	50	48	41	29	21	9.2

TABLE III
Effects of Poly(α -terpineol-*co*-styrene) on the IPN Properties

Sample	Poly(α -terpineol- <i>co</i> -sty) (base M) $\times 10^2 \text{ mol}^{-1}$	Yield (%)	Swelling in DMF (%)	M_c in DMF
IPN-1	0.05	24	58	857
IPN-2	1.10	37	69	1160
IPN-3	1.66	48	85	1445
IPN-4	2.21	56	95	1785
IPN-5	3.32	75	121	2563

[BMA] = 0.89 mol/L; [AIBN] = 5.23×10^{-3} mol/L; [DVB] = 0.76 mol/L. temperature = $85 \pm 0.1^\circ\text{C}$; time = 1 h.

TGA. The TGA curve of poly(butyl methacrylate) (Fig. 8) exhibited weight loss with temperature. The polymer was stable up to 200°C . The thermal behavior data were as follows:

1. The onset of major weight loss occurred at 249°C ; the completion of major weight loss occurred at 331°C .
2. The polymer was almost volatilized around 289°C .

Synthesis of simultaneous IPNs

A solution of poly(α -terpineol-*co*-styrene)_(base M) (0.05 – 3.32×10^2 mol/L), BMA (0.36 – 2.15 mol/L), AIBN (1.74 – 10.45×10^3 mol/L), and DVB (0.25 – 1.56 mol/L) in xylene was polymerized for 1 h at $85 \pm 0.1^\circ\text{C}$ under an inert atmosphere of nitrogen. The IPNs formed were precipitated in methanol and vacuum-dried until a constant weight was obtained. Sixteen IPNs were prepared with the variables shown in Table I. The principal chemical variables included a systematic variation of concentrations of poly(α -terpineol-*co*-styrene), BMA, AIBN, and DVB.

The formation mechanism of the IPNs is shown in Scheme 1.

TABLE IV
Effect of BMA Concentration on the IPN Properties

Sample	[BMA] (mol/L)	Yield (%)	Swelling in DMF (%)	M_c in DMF
IPN-6	0.36	68	94	1700
IPN-3	0.89	48	85	1445
IPN-7	1.25	41	69	1223
IPN-8	1.79	32	51	945
IPN-9	2.15	23	48	695

Poly(α -terpineol-*co*-styrene)_(base M) = 1.66×10^{-2} mol/L; [AIBN] = 5.23×10^{-3} mol/L; [DVB] = 0.76 mol/L. Temperature = $85 \pm 0.1^\circ\text{C}$; time = 1 h.

TABLE V
Effect of AIBN Concentration on the IPN Properties

Sample	[AIBN] $\times 10^3$ (mol/L)	Yield (%)	Swelling in DMF (%)	M_c in DMF
IPN-10	1.74	32	71	985
IPN-3	5.23	48	85	1445
IPN-11	8.71	54	99	1823
IPN-12	10.45	62	105	2065

Poly(α -terpineol-*co*-styrene)_(base M) = 1.66×10^{-2} mol/L; [BMA] = 0.89 mol/L; [DVB] = 0.76 mol/L. Temperature = $85 \pm 0.1^\circ\text{C}$; time = 1 h.

Characterization of the IPNs

Before we proceeded to measure the swelling of the polymer networks in different solvents, the soluble or uncrosslinked component of the IPNs was removed with the help of a Soxhlet extractor. The percentage extractable material was calculated as follows:

$$\text{Extractable material (\%)} = \frac{W_b - W_a}{W_a} \times 100$$

where W_b is the weight of the IPN before extraction and W_a is the weight of the IPN after extraction.

Swelling measurements

The crosslink density in polymer networks is usually estimated through solvent absorbency (swelling) measurements. We calculated the swelling data by soaking the sample in different polar and nonpolar solvents, such as dimethylformamide (DMF), dimethyl sulfoxide (DMSO), dioxane, benzene, and toluene, until an equilibrium weight was achieved (~ 24 h). We made weight measurements by blotting the samples dry and immediately weighing them. We then removed the swelling solvent by heating the samples to 60°C *in vacuo* until an equilibrium weight was achieved. The percentage swelling was calculated according to the following relationship:⁴⁰

TABLE VI
Effect of DVB Concentration on the IPN Properties

Sample	[DVB] (mol/L)	Yield (%)	swelling in DMF (%)	M_c in DMF
IPN-13	0.25	25	102	1732
IPN-14	0.51	33	94	1565
IPN-3	0.76	48	85	1445
IPN-15	1.02	55	66	1223
IPN-16	1.56	65	50	1120

Poly(α -terpineol-*co*-styrene)_(base M) = 1.66×10^{-2} mol/L; [BMA] = 0.89 mol/L; [AIBN] = 5.23×10^{-3} mol/L. Temperature = $85 \pm 0.1^\circ\text{C}$; time = 1 h.

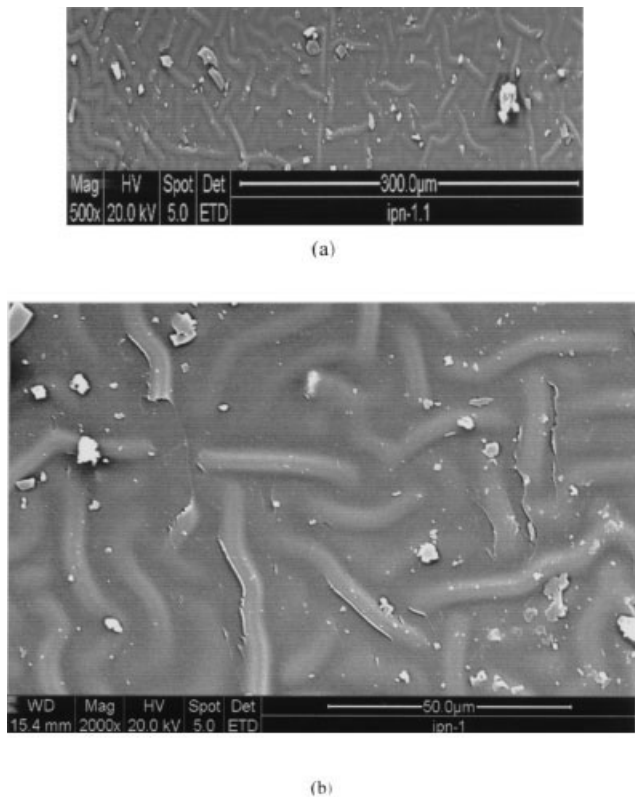


Figure 10 SEM photograph of IPN-3 at magnifications of (a) 500 and (b) 2000 \times .

$$\text{swelling (absorbency; \%)} = \frac{W_s - W_d}{W_d} \times 100$$

where W_s is the weight of the swollen IPN and W_d is the weight of the dry IPN.

Crosslink density

The crosslink density of the network was determined with the swelling data of the IPNs in DMF with the help of the Flory–Rehner equation:^{41,42}

$$\frac{1}{M_c} = - \frac{\ln(1 - V_p) + V_p + X_{12}V_p^2}{\rho V_1(V_p^{1/3} - V_p/2)}$$

where M_c is the average molecular weight between crosslinks of the network, ρ is the density of the network, V_1 is the molar volume of the solvent, V_p is the volume fraction of the polymer in the swollen gel, and X_{12} is the polymer–solvent interaction parameter calculated by the following expression.⁴³

$$X_{12} = B + \frac{V_1}{RT} (\delta_p - \delta_s)^2$$

where δ_p and δ_s are the solubility parameters⁴³ of the polymer and the swelling solvent, respectively; R is the gas constant; T is the temperature; and B is the lattice constant, the value of which was taken as 0.34.⁴¹

Activation energy (E_a)

E_a of IPN synthesis was calculated from the Coats and Redfern equation:⁴⁴

$$\log[g(\alpha)/T^2] = \frac{E_a}{2.3 RT} + \log[ZA/B \Delta E(1 - 2RT/E_a)]$$

$$g = - \log[(1 - \alpha)/n]$$

where Z is the preexponential factor, B is the linear heating rate, α is the degree of cure, n is the order of reaction, A is the Arrhenius constant, and ΔE is the apparent activation energy.

The plot of $\log[g(\alpha)/T^2]$ versus $1/T \times 10^3$ gave a linear curve whose slope was equal to $-E_a/2.3RT$, and its intercept corresponded to $\log_{10}[ZA/B \Delta E(1 - 2RT/E_a)]$.

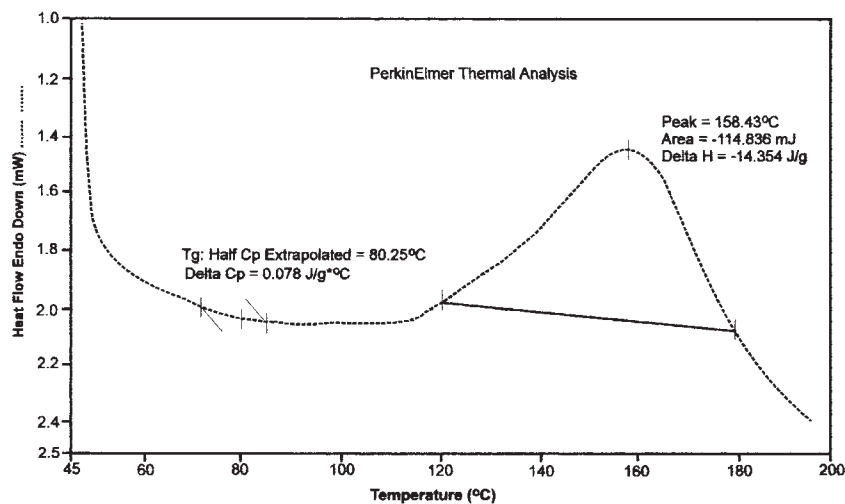


Figure 11 DSC curve of IPN-3 heated from 40 to 200°C at 10°C/min.

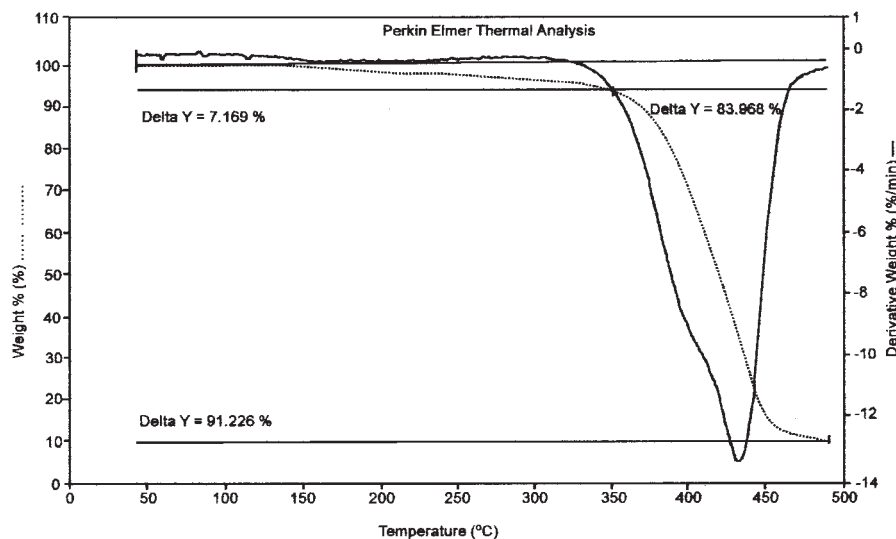


Figure 12 TGA curve of IPN-3 heated from 40 to 500°C at 10°C/min.

RESULTS AND DISCUSSION

Structural evidence for the IPN was determined from the Fourier transform infrared (FTIR) spectrum (Fig. 9), which indicated bands at 1723 cm^{-1} for ester groups of BMA, 3430 cm^{-1} for alcoholic groups of α -terpineol, and 3010 cm^{-1} for aromatic rings.

Emphasis is given to the effect of the concentrations of poly(α -terpineol-*co*-styrene), BMA, AIBN, and DVB on properties such as swelling, crosslink density, and phase behavior.

The results of swelling in various solvents and the extraction study with DMF are summarized in Table II. The low percentage of extractable material shown in Table II may have been due to the uncrosslinked portion remaining within the matrix. This uncrosslinked part may have been comprised of some graft and/or linear copolymer.

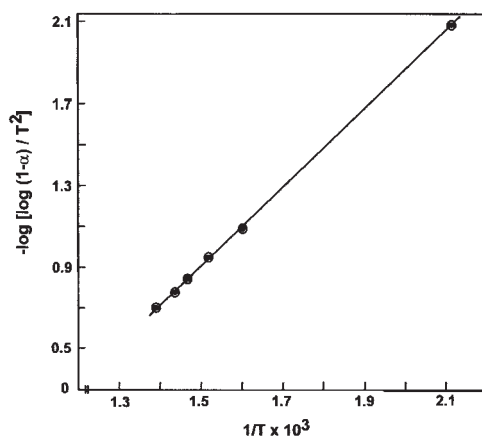


Figure 13 Graph plotted between $-\log[\log(1 - \alpha)/T^2]$ and $1/T \times 10^3$.

Effect of composition

The effect of the composition of the copolymer followed a logical trend because an increase in the concentration of α -terpineol-*co*-styrene_(base M) copolymer resulted in increased swelling and M_c (Table III). This implies that the presence of the copolymer restricted the crosslinking of BMA. As the concentration of the α -terpineol-*co*-styrene copolymer increased, the probability of crosslinking between the two polymers increased. A similar explanation could be given for the fact that the percentage of swelling and M_c were inverse functions of the concentration of BMA (Table IV).

Effect of the AIBN initiator

Table V presents data on the effect of the concentration of AIBN on the swelling and M_c of IPN. The data showed that both swelling and M_c increased with increasing concentration of AIBN.

Effect of the DVB crosslinker

Table VI shows that an increase in the concentration of DVB increased the percentage yield, as expected. The reason for this may have been that the increased crosslinking agent increased crosslinking and, consequently, the percentage yield. The percentage swelling of the IPNs in different solvents and M_c were inverse functions of the concentration of DVB. The explanation is that an increase of the concentration of DVB increased the crosslinking between the two polymeric networks, which resulted in decreased swelling and M_c .

Morphology and thermal properties

We used SEM to examine surface morphology. The SEM study revealed the morphology exhibited by the IPNs (Fig. 10) at a different magnifications, which indicated a threadlike network structure. This observation was a consequence of the DSC results that showed the T_g for IPN at 80.2°C. The exotherm peak temperature 158.43°C indicated network formation within the system (Fig. 11).

TGA

An IPN sample (Fig. 12) containing α -terpineol-*co*-styrene and poly(butyl methacrylate) was stable up to 350°C and started losing weight above this temperature. Rapid decomposition was observed at 350–450°C, and the near volatilization of the IPN was observed around 490°C. A total weight loss of 91.23% was observed in the range 40–500°C. The weight losses at different stages of temperature were as follows:

1. 40–350°C = 7.16%.
2. 350–500°C = 83.96%.

E_{ar} , calculated with the help of the Coats and Redfern equation from the slope of the linear graph of $-\log[\log(1 - \alpha)/T^2]$ versus $1/T \times 10^3$ (Fig. 13), was 23 kJ/mol.

CONCLUSIONS

The major conclusions drawn were as follows:

1. M_c was a direct function of the copolymer and initiator concentrations, whereas it was an inverse function of the monomer and crosslinker concentrations.
2. The IPNs exhibit characteristic morphologies, which indicated a threadlike network structure.
3. The IPNs showed good thermal properties.

The authors thank K. P. Singh, Director, Harcourt Butler Technological Institute, Kanpur, India, for providing the necessary facilities and the Advanced Center of Material Sciences, IIT, Kanpur, India, for providing the DSC and TGA. The authors also thank Jitendra Kumar, Material Science Programme, IIT, Kanpur, India, for his cooperation.

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