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ORIGINAL PAPER

Simultaneous Interpenetrating Polymer Networks of Polyurethane from Pentaerythritol–Modified Castor Oil and Polystyrene: Structure–Property Relationships

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Abstract Castor oil was transesterified with pentaerythritol thereby introducing reactive hydroxyl groups. The method of matrix-assisted laser desorption/ionization time-of-flight mass spectroscopy was used to determine the components of the castor oil pentaerythritol alcoholysis products. Simultaneous interpenetrating polymer networks of pentaerythritol modified castor oil and diphenyl-methane 4-4′ diisocyanate polyurethane (P1-CO-PU) and polystyrene (PS) were synthesized using benzoyl peroxide as the initiator and divinyl benzene as the crosslinker. The effect of PU/PS composition on the morphology, miscibility and physical properties of the resulting IPNs was investigated by scanning electron microscopy, dynamic mechanical thermal analysis, measurements of mechanical properties and resistance to chemical reagents. The patterns of the extent of the phase separation and the characteristics of the interpenetration reached as a function of the components were deduced.

Keywords Pentaerythritol-modified castor oil · Polyurethane · Interpenetrating polymer networks (IPN) · Structure–property relation

Introduction

Castor oil is a renewable resource and a natural polyol and has been a successful candidate in the formation of industrially useful materials like cost-effective polyurethane elastomers, it is yet to be exploited for the modification of polymers [1–3]. The polyurethanes obtained from castor oil are typically water resistant and flexible due the presence of a long fatty acid chain and lend themselves to thermosetting-type structures due to their trifunctional nature. The long pendant chains of the fatty acids impart flexibility and hydrolysis resistance to the network and the double bond present in the castor oil serves as a grafting center. The properties of materials obtained from castor oil include low hydroxyl number leading to inherently low modulus materials, a sluggish rate of curing of secondary hydroxyl groups and structural irregularity due to steric hindrances caused by the long pendant fatty acid chains during polyurethane formation, resulting in low tear strength [4–6].

Oil-based polyurethanes are mixed in the form of interpenetrating networks with others monomers like styrene, methacrylate, formaldehyde, etc. in order to modify these properties. Castor oil is also transesterified or alcoholized with alcohols thereby introducing reactive hydroxyl groups. Most of the literature published has illustrated the use of glycerol for transesterification.

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However, glycerol (a component of the triglyceride molecule) is readily destructible at high temperatures. This disadvantageous property stems from the presence of hydrogen atoms in position β relative to the hydroxyl group in the glycerol molecule. This structural feature is conductive to the partial defragmentation of the molecule and the formation of unsaturated compounds. The problem can be solved by replacing glycerol with another polyhydric alcohol that does not contain β-hydrogen atoms, i.e. pentaerythritol [7, 8].

Though voluminous literature is available on modified castor oil polyurethane, the study of IPNs of modified castor oil polyurethane has not been paid due attention [7, 9–13]. In the present study a family of simultaneous IPNs based on pentaerythritol modified castor oil and diphenylmethane 4–4′ diisocyanate polyurethane (P-PU) and polystyrene (PS) were synthesized using benzoyl peroxide (PBO) as the initiator and divinyl benzene (DVB) as the crosslinker has been studied. The goal of this work was to study the effect of polyol modification on the mechanical, chemical and rheological properties of the resulting simultaneous interpenetrating polymer networks (SINs), as a function of the percentage of modifier agent (pentaerythritol). The properties of the IPNs were studied related to their chemical structure and morphology. The morphology of IPN was investigated as related to compatibility and microphase segregation between the polyurethane from modified castor oil and polystyrene. Thermomechanical properties of the synthesized IPNs were analyzed using a dynamic mechanical thermal analyzer (DMTA). The DMTA study of the relaxation associated with the glassy transition of the two components of the IPN, the patterns of the extent of the phase separation and the characteristics of the interpenetration reached as a function of the components were deduced.

Experimental Procedure

Materials

Castor oil (CO) was of commercial grade and purchased from the local market (Bucaramanga, Colombia). It was dried at 105 °C under a nitrogen atmosphere and characterized for its hydroxyl value (160 mg KOH/g), acid value (2 mg KOH/g) and ricinoleic acid content of 90%. Diphenyl-methane-4,4′-diisocyanate (MDI) was procured from E-Merck. Styrene (S) and DVB were used freshly distilled after washing with 5% NaOH solution. Benzoyl peroxide (BPO) was used as the radical initiator for styrene (Aldrich, Germany).

The polystyrene–polyurethane IPNs studied in this work contained two parts, the polyurethane phase and the polystyrene phase, designated as SINPS*x*PU*y* (*x* stands for the % weight of polystyrene and *y* the % weight of polyurethane).

Transesterification Castor Oil Using Pentaerythritol

A resin kettle equipped with a stirrer, reflux condenser and nitrogen inlet was charged with 400 g of castor oil and pentaerythritol in a previously established proportion, along with a catalyst litharge (lead(II) oxide-PbO) 0.05% based on the oil weight. The reaction was carried out at 200 °C for 2.5 h. The resultant polyol was dried at 80 °C under vacuum. The method of matrix-assisted laser desorption/ionization time-of-flight mass spectroscopy (MALDI TOF MS) was used to determine the components of castor oil pentaerythritol alcoholysis product. MALDI TOF measurements were performed with an ABI 4700 TOF-TOF MALDI TOF (time-of-flight) mass spectrometer. The acceleration voltage used was 25 kV. The positive ions were detected with reflectron mode (20 kV). The nitrogen laser operating at 4 Hz was used to produce laser desorption. The sample was dissolved in dimethyl formamide (DMF) with a concentration of 30 mg/mL, and matrix material 2,5-dihydroxybenzoic acid (DHB) was also dissolved in DMF. The sample solution and matrix solution were mixed in a ratio of 5:2 and electrolyte NaCl solution was added. Of these solutions, 0.5–1 µL was deposited onto a sample plate (stainless steel) and allowed to air dry [1].

Synthesis of Pentaerythritol Modified Castor Oil Polyurethane

The reaction was carried out in a five-necked, 500-ml, round-bottom flask fitted with an agitator, a temperature indicator and nitrogen inlet-out tubes. The flask was kept in a water bath maintained at the desired temperature. A calculated amount of moisture-free pentaerythritol modified castor oil was taken in the flask and a corresponding amount of MDI (NCO/OH = 1) was added gradually so that the complete the addition of MDI took 5–10 min, and cured at room temperature for 4 h. After curing the material was left in a heated oven at 90 °C for 8 h.

Synthesis of Simultaneous Interpenetrating Polymer Networks (SINs)

The castor oil modified and MDI was thoroughly mixed with the styrene. The initiator BPO and the DVB (1.4% and 2% by weight of styrene, respectively) as well as the catalyst dibutyl tin dilaurate (DBTL; 0.5% by weight of polyol) were added. As the mixture became viscous, it was poured into the mold and kept under pressure and the rest

Table 1 Data on composition of different samples of SINs and hydroxyl value of castor oil based polyols

Sample Identification	Polyol	Concentration of during transesterification (mass pentaerythritol/ mass castor oil)	Hydroxyl value (mg KOH/g sample)
SIN-CO-PS0PU100	CO	0	160
SIN-CO-PS10PU90			
SIN-CO-PS20PU80			
SIN-CO-PS30PU70			
SIN-CO-PS40PU60			
SIN-P0-CO-PS0PU100	P0	1.3%	192
SIN-P0-CO-PS10PU90			
SIN-P0-CO-PS20PU80			
SIN-P0-CO-PS30PU70			
SIN-P0-CO-PS40PU60			
SIN-P1-CO-PS0PU100	P1	2.6%	236
SIN-P1-CO-PS10PU90			
SIN-P1-CO-PS20PU80			
SIN-P1-CO-PS30PU70			
SIN-P1-CO-PS40PU60			
SIN-P2-CO-PS0PU100	P2	5.3%	250
SIN-P2-CO-PS10PU90			
SIN-P3-CO-PS20PU80			
SIN-P4-CO-PS30PU70			
SIN-P5-CO-PS40PU60			

of the reactions were allowed to proceed in the mold. It was kept at room temperature for 24 h and at 120 °C for 8 h. A series of IPNs of different compositions were obtained following the same procedure (Table 1). The finished sheets were cut in desired shapes and sizes for further study.

Characterization

Chemical and solvent resistances were measured according to the ASTM D543-67 method. Each polymer specimen of known dimensions was immersed in solvents with increasing order of solubility parameter δ from 8.2 to 14.5 (cal/cm^3)$^{1/2}$. This procedure was continued till equilibrium swelling was achieved. The swelling behavior of the polyurethanes in various solvents was investigated. The crosslink density of polyurethanes was determined according to the rubber elasticity theory (considering the front factor to be unity). The variation of the crosslink density of polyurethanes was studied based on the hydroxyl value of the polyol utilized in the synthesis. The stress-strain properties were determined using an Instron universal testing machine according to ASTM D-527 procedure and hardness measurements were made on a Shore-A hardness test apparatus using ASTM-785 method. DMTA of the selected polyurethanes were carried out on a Rheometrics DMTA V. The samples were run with a temperature scan from −150 to 170 °C at a frequency of 1 Hz. The heating rate for all the runs was 5 °C/min. A nitrogen purge of 60 mL/min was used. All the analyzed samples had approximately the same dimensions of 2 cm × 0.6 cm × 0.3 cm.

The storage modulus (E') and loss factor (tan δ) were measured. The temperature corresponding of the maximum of the tan δ peak position was taken as glass transition temperature (T_g). Scanning electron micrographs were obtained using a JEOL JSM-6400 electron microscope. The samples had been previously coated with a gold layer and the morphology of the materials was studied through their fractures after immersion into liquid nitrogen [9, 10].

Results and Discussion

Characterization of Castor Oil Transesterified with Pentaerythritol

The hydroxyl values of polyols (P0-CO [molar ratio pentaerythritol/castor oil = 0.1], P1-CO [0.2] and P2-CO [0.4]) obtained from different concentrations of pentaerythritol used in the castor oil transesterification are given in Table 1.

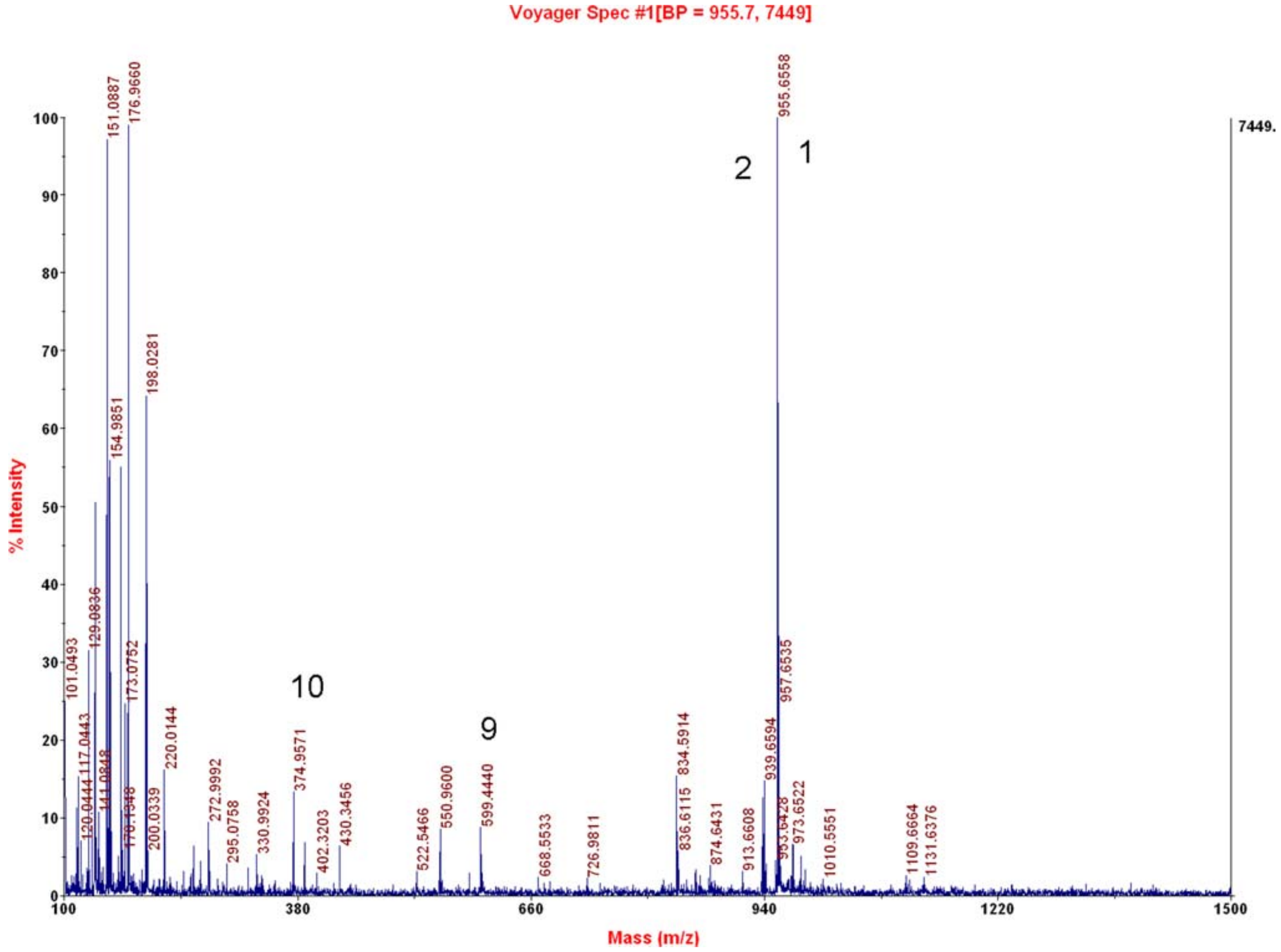

Fig. 1 MALDI TOF mass spectra of castor oil (CO) for the range 100–1,500 *m*/*z*

Table 2 Individual components identified in castor oil (CO) on MALDI TOF mass spectroscopy

Number	Structure	Observed (*m*/*z*)	Calculated (*m*/*z*)	Intensity
1	tri-ricinolein +Na + H	957.649	956	0.79
2	tri-ricinolein + Na	955.6479	955	0.95
3	tri-ricinolein + Na + H−H_2O	939.6531	938	0.165
4	tri-ricinolein + Na + H_2O	937.6385	937	0.28
5	tri-ricinolein −H_2O	913.6388	914	0.08
6	tri-ricinolein + H−$3H_2O$	879.6540	879	0.03
7	di-ricinolein +Na	675.4475	675	0.05
8	di-ricinolein + H−$2H_2O$	617.4396	617	0.05
9	di-ricinolein + H−$3H_2O$	599.4442	599	0.17
10	mono-ricinolein + H	374.9571	373	0.14
11	Stearic acid + Na−$2H_2O$	272.9992	271	0.085
12	Palmitic acid −$2H_2O$	220.0144	220.4	0.15

In Fig. 1 MALDI TOF mass spectrum of castor oil, the main peak appears at 955 *m*/*z*, representing triricinolein (triglyceride of ricinoleic acid), the most abundant triacylglycerol of castor oil. Two peaks in the mass range at 600 y 675 *m*/*z* were detected, referring to diacylglycerol of castor oil. A very low intensity peak at 374 *m*/*z* refers to monoacylglycerol of castor oil. Table 2 illustrates the observed mass and calculated mass of all components as identified by MALDI TOF mass spectroscopy for castor oil. To calculate mass fractions of various components,

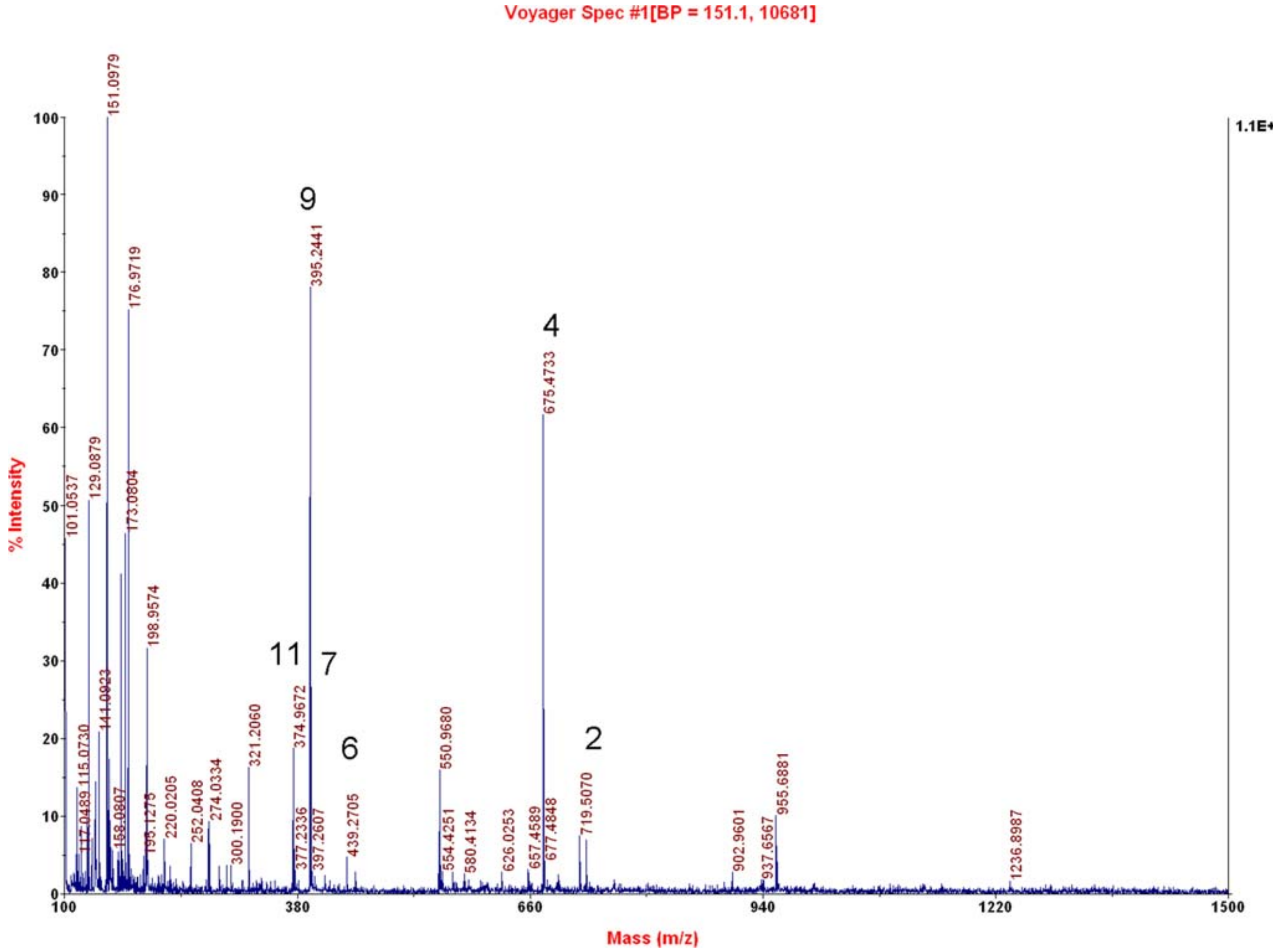

Fig. 2 MALDI TOF mass spectra of pentaerythritol modified castor oil (P1-CO) for the range 100–1,500 *m*/*z*

total peak intensity was calculated for each component (by adding peak intensities of the same component shown at different *m*/*z* value, as identified in Table 2). Mass fractions of various components present in castor oil as calculated using MALDI MS are: triacylglycerol (77%), diacylglycerol (14%) and 9% nonfunctional acid residues.

In the MALDI TOF MS spectrum for pentaerythritol modified castor oil (P1-CO) polyol, Fig. 2, appears a very low intensity peak at 955 *m*/*z*. In contrast, the peaks corresponding to diacylglycerol and monoacylglycerol at 675 and 395 *m*/*z*, respectively, grew in intensity. There are two peaks of medium intensity at 719 and 411 *m*/*z*, referring to diricinoleate of pentaerythritol and monoricinoleate of pentaerythritol. Medium intensity peaks at 439, 403 and 344 *m*/*z* represent monoricinoleate of pentaerythritol. Table 3 illustrates the observed mass and calculated mass of all components as identified by MALDI TOF mass spectroscopy for P1-CO. It can argue, based on the results, that castor oil reacted by transesterification with pentaerythritol and there was a mixture that contains mostly monoricinoleate of pentaerythritol and monoacylglycerol. Diricinoleate of pentaerythritol and diacylglycerol are present in minor proportion. Mass fractions of various components present in P1-CO as calculated using MALDI MS are: diacylglycerol (20.8%), monoacylglycerol (40.5%), diricinoleate of pentaerythritol (8.5%), monoricinoleate of pentaerythritol (21.2%) and 9% nonfunctional acid residues [1].

Characterization of IPNs

The FTIR spectra of all the SINs showed characteristic peaks in the wave number region 3,450–3,200 cm^{-1} and 1,750–1,650 cm^{-1}, which correspond to secondary urethane >N–H and >C=O stretching groups. All IPNs medium weak peaks of aromatic bonds stretching between 1,600 and 1,450 cm^{-1}, and absorption characteristic peak at 750 cm^{-1} indicate the presence of phenyl groups of the PS. All these observations revealed that there are not collateral reactions between the polyurethane and polystyrene.

Table 3 Individual components identified in pentaerythritol modified castor oil (P1-CO) on MALDI TOF mass spectroscopy

Number	Structure	Observed (*m/z*)	Calculated (*m/z*)	Intensity
1	tri-ricinolein + Na	956.6736	955	0.03
2	di-ricinoleate of pentaerythritol + Na	719.4822	719	0.1
3	di-ricinoleate of pentaerythritol + Na + H	720.5450	720	0.05
4	di-ricinolein + Na	675.4583	675	0.5
5	di-ricinolein + H$-$H$_2$O	657.4527	657	0.05
6	mono-ricinoleate of pentaerythritol + Na	439.2469	439	0.06
7	mono-ricinoleate of pentaerythritol	411.2016	416	0.12
8	mono-ricinoleate of pentaerythritol + Na$-$2H$_2$O	405.0971	403	0.05
9	mono-ricinolein + Na	395.2292	395	0.99
10	mono-ricinolein + H	374.9695	373	0.08
11	mono-ricinoleate of pentaerythritol + Na$-$4H$_2$O	342.6783	344	0.05
12	Stearic acid + Na$-$2H$_2$O	272.9992	271	0.085
13	Palmitic acid $-$2H$_2$O	220.0144	220.4	0.15

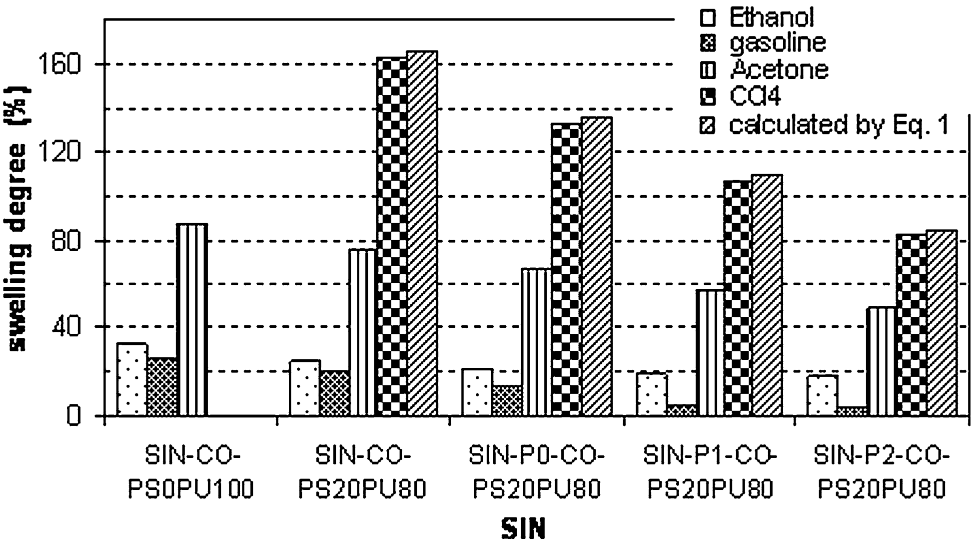

Fig. 3 Swelling degree of various chemicals in IPN sheets (experimental data and calculated data according to Eq. 1)

This means that the IPNs formed do not posses chemical bonds between the two networks [12].

The percentage weight losses of IPNs were determined in NaOH·H$_2$O, HCl·H$_2$O and CH$_3$COOH. All the IPNs show excellent acid and alkali resistance as compared to unmodified castor oil polyurethane/polystyrene IPNs. It was observed that IPNs irrespective of NCO/OH ratio and PU/PS composition were stable in acid and alkali, whereas in ethanol, acetone and chloroform (CCl$_4$) IPNs showed a varying amount of swelling. IPNs obtained from castor oil (CO-IPNs) showed relatively high swelling as compared to that of IPNs obtained from pentaerythritol modified castor oil (P0-CO, P1-CO and P2-CO IPNs), due to the fact that pentaerythritol modification results in more crosslinking and stiffer IPNs (Fig. 3) [3–5].

To confirm the formation of the IPN structure, the IPN samples were cut into small pieces, and the weighed quantity (about 10 g) was packed in filter paper for each case. The packets were kept in a Soxhlet extraction column and extracted with toluene for 24 h. The packets were dried at 80 °C for 24 h in an oven, cooled in a desiccator, and weight again to find the soluble content. Toluene extraction showed a sol component less than 6% for all IPNs synthesized. Both of the above results support the formation of IPN structures.

The theoretical swelling degree of the IPN was calculated from the swelling data of the individual networks, using the swelling degree by weight, rather than by volume, because of weight-conservation between individual polymers and polymers in the IPN form by the relation [13]:

$$\frac{1}{A_{\mathrm{ipn}}} = \frac{w_1}{A_1} + \frac{w_2}{A_2}. \tag{1}$$

where w_1 and w_2 are the weight fractions of each network in the dry state, A_1 and A_2 are the swelling degrees of the individual networks, respectively, and A_{ipn} is the swelling degree of the IPN. In this case network 1 and network 2 are mixed in the IPN form with two co-continuous pure phases. In the presence of the swelling solvent, both networks swell mutually. That equation does not involve any kind of physical crosslinks or mutual entanglements. Hence, if molecular interpenetration were to exist for IPNs, the experimental swelling value would be lower than the value calculated from Eq. 1. This is only true if there is no possibility of chemical grafting between the networks. Note also that the contribution of the polymeric species to the swelling of one network can be neglected with regard to the swelling behavior of the molecular solvent. To measure the equilibrium swelling degree, preweighed dry samples were immersed in CCl_4, a common solvent for both polymers. The calculated swelling degrees of PU/PS IPNs according to Eq. 1 are close to the experimental swelling degree of IPNs synthesized (see Fig. 3). This demonstrates the absence of additional chemical interactions for IPNs, and agrees with the results of FTIR [14].

Figure 4 shows the mechanical properties of SINs obtained from P2-CO, P1-CO and CO with varying amounts of styrene. It is observed that pentaerythritol modified castor oil polyurethane and polystyrene interpenetrating polymer networks (P2-IPNs and P1-IPNs) exhibited better mechanical properties as compared to unmodified castor oil polyurethane/polystyrene IPN (CO-IPNs), due to more crosslinking density. The tensile strength and Shore A hardness increase up to 20% styrene, and starts to decrease above 20% styrene. The change in the properties below 20% styrene indicates that there is little intermixing between the two polymers, and that styrene is dispersed in the polyurethane continuous phase. The steep rate of decrease in the properties beyond styrene concentration of 25% implies phase inversion (this drop in the properties suggest that phase inversion occurs at about 75%PU/25%PS, where the continuous matrix changes from the elastomeric PU to the rigid PS) [11].

Figure 5 shows the curves of storage modulus E' and tanδ versus temperature for (P1-CO)-IPNs with different PU/PS compositions. It can be seen from Fig. 5a that when the PS content increases the rigidity of the system increases. Data of the glass transition temperatures (T_g) obtained by the maxima in the tan delta curves (Fig. 5b) together with the predictions of the Wood equation (Eq. 2) for P1-IPNs are summarized in Table 4. The appearance of single T_g indicated the formation of the interpenetrating polymer networks. With an increase in PS concentration in the IPN the T_g of the IPN shifted towards the T_g of PS.

$$T_g = w_1 T_{g1} + w_2 T_{g2} \tag{2}$$

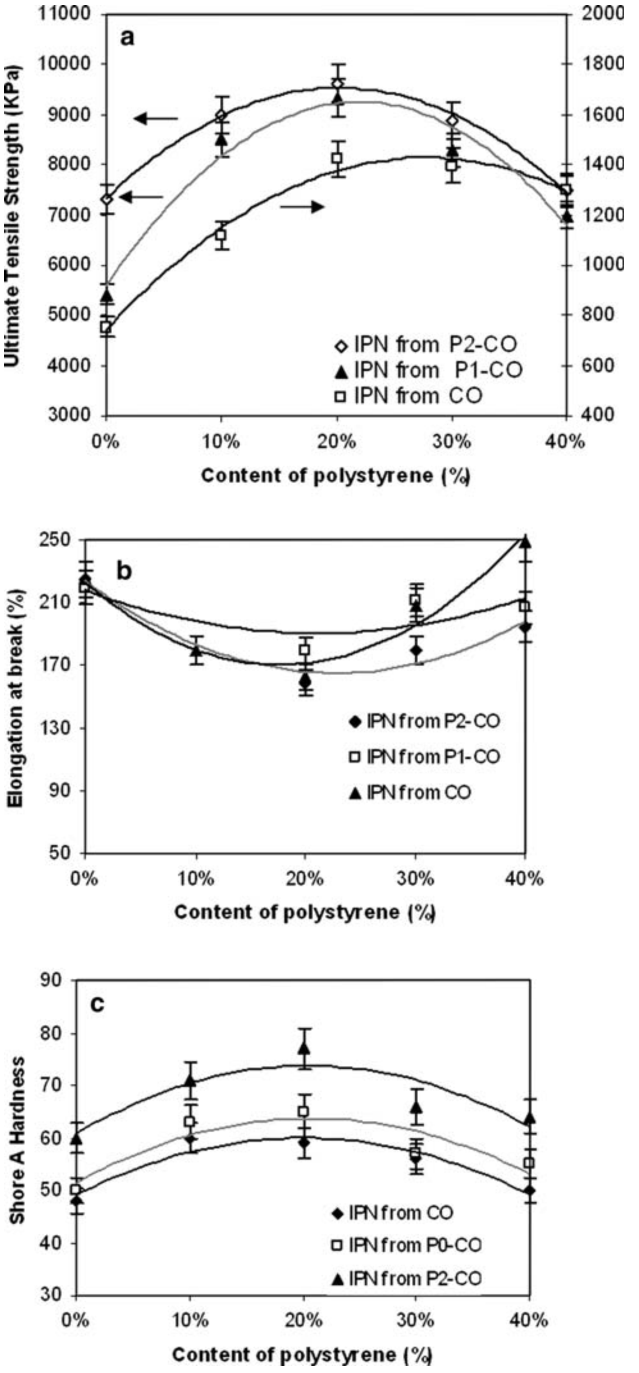

Fig. 4 Mechanical properties of IPNs. **a** Ultimate tensile strength, **b** elongation % at break and **c** shore A hardness

where w_1 and w_2 are the weight fractions of each network, T_{g1} and T_{g2} are the glass transition temperatures of the individual networks, respectively, and T_g is the glass transition of the IPN (Table 4).

In order to calculate the compatibility between the networks, the compatibility factor (θ) was calculated from a theoretical equation of DiBeneditto modified by Xiao et al. using Eq. [3]:

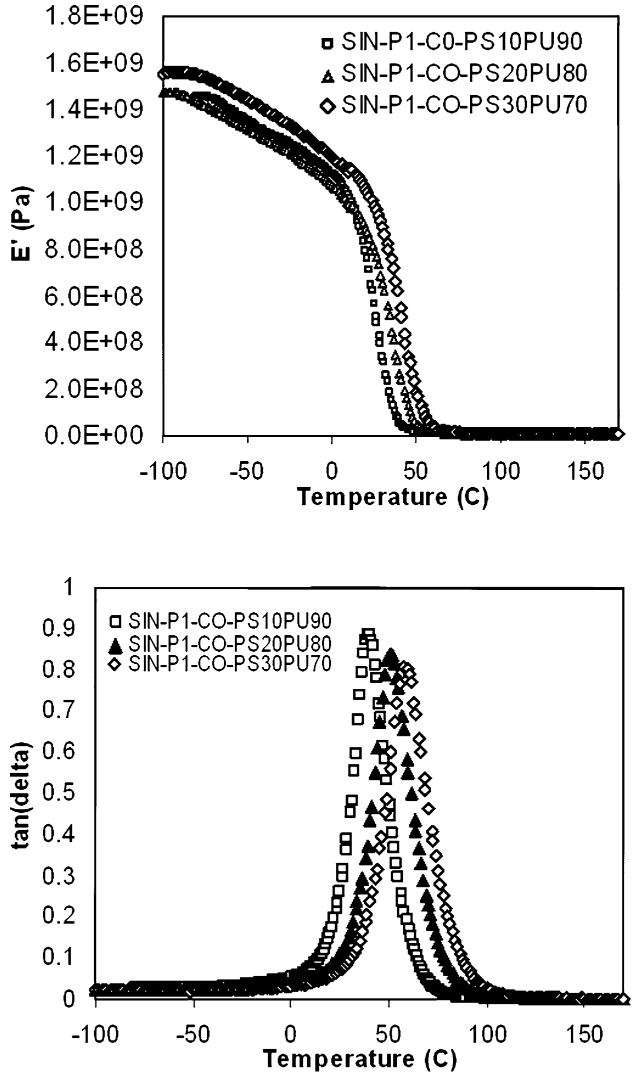

Fig. 5 Dynamo-mechanical curves of IPN based on P1-CO-PU/PS with different PU/PS composition **a** module of storage E' and **b** tan δ

$$\left[T_{\mathrm{g}}(\mathrm{calcd}) - T_{\mathrm{g}}(\mathrm{DMTA})\right]/T_{\mathrm{g}}(\mathrm{calcd}) = \frac{-\theta}{(1+\theta)} \tag{3}$$

where θ is the compatibility factor, $T_{\mathrm{g(calcd)}}$ and $T_{\mathrm{g(DMTA)}}$ are the glass transition temperature calculated with Wood equation and obtained experimental by DMTA, respectively. A decrease in θ implies greater compatibility caused by increasing physical entanglements produced by a partial interpenetration between the networks. The system P1-CO-PU/PS shows limited compatibility at low concentrations of PS ([PS] $< 20\%$) and full incompatibility at higher concentrations of PS ([PS] $> 30\%$). The change in the compatibility factor in the given composition interval are connected with the phase inversion. The higher is the phase separation, the smaller is the contribution of entanglements to the effective network density.

Network crosslinking density (v_{e}) was calculated by the following equation, derived from the theory of rubber elasticity [8]:

$$E' = 3v_{\mathrm{e}}(RT) \tag{4}$$

where E' is the storage modulus of the thermoset in the rubbery plateau region at $T_{\mathrm{g}} + 30$ °C, R is the gas constant, and T is the absolute temperature. In the case of CO-IPNs, lower crosslinking density may be caused by incomplete chemical reaction or cyclization reactions during crosslinking (Table 4). The probability of the formation of elastically inactive network structures during formation of the polyurethane increases with increasing functionality of the polyols. The values of v_e for the IPN were higher as compared to the primary network. This effect was explained by the contribution of the mechanical entanglements between networks.

The morphology of P1-CO-PU/PS IPNs for various compositions is shown in Fig. 6. The formation of IPN structure is vividly seen by the existence of a finely mixed microstructure of polyurethane and polystyrene. The electron micrographs of IPN show phase separation with polyurethane one phase (black) and polystyrene the other phase (white). Although all the samples are transparent, for some intermediate formulations, the existence of two domains is evident, which can be attributed to separate polyurethane and polystyrene phases. The data on the microphase structure of the IPN show that the phase separation proceeds according to the spinodal mechanism in spite of the simultaneously proceeding chemical reaction. The structure is dependent on the concentration ratio of two networks and, depending on this ratio, phase

Table 4 Glass transition temperatures (T_{g}) and crosslinking density (v_{e}) of modified castor oil and unmodified castor oil polyurethane/polystyrene IPN systems

Sample identification	Glass transition temperature (°C)		Compatibility factor (θ)	Crosslinking density v_{e}(mol/m^3)
	By DMTA	Calculated using Wood equation		
SIN-CO-PS20PU80	48 ± 2	43	0.130	191
SIN-P1-CO-PS10PU90	50 ± 2	42	0.117	262
SIN-P1-CO-PS20PU80	52 ± 2	48	**0.077**	**270**
SIN-P1-CO-PS30PU70	60 ± 2	55	0.092	258

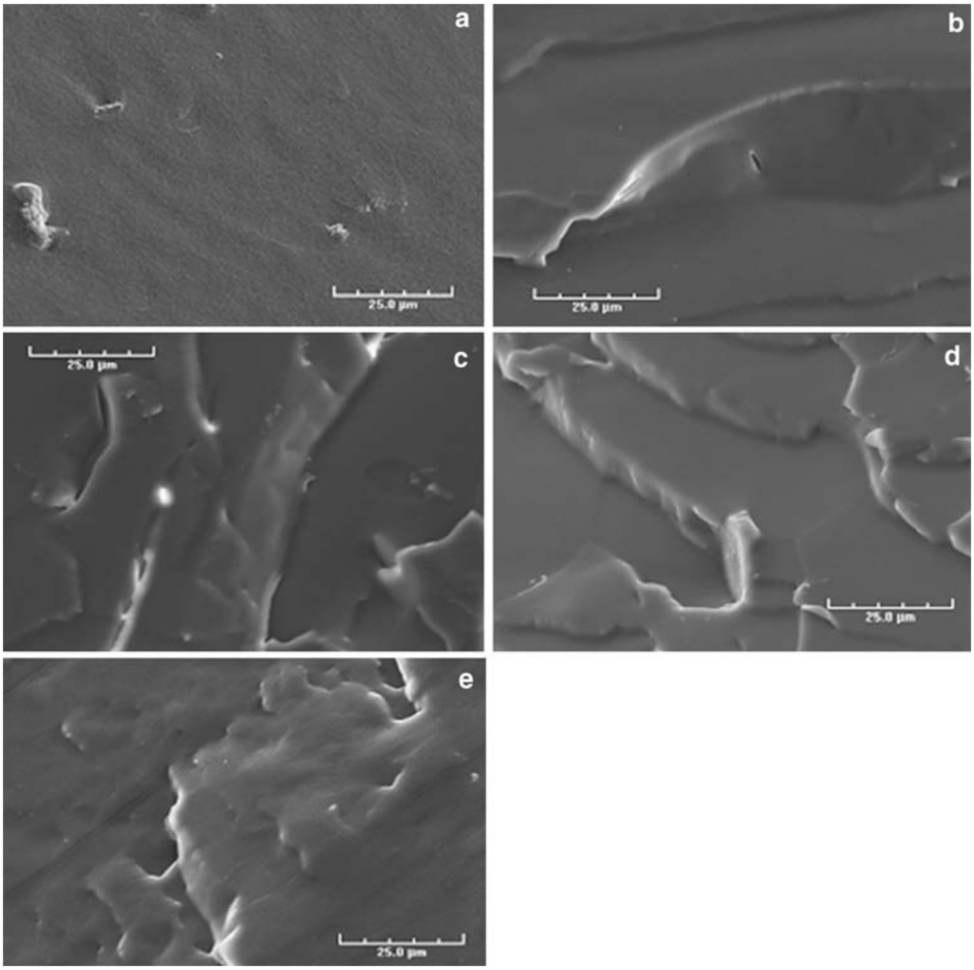

Fig. 6 Scanning electron micrographs of SINs prepared from polyol P1, with various levels of polystyrene at NCO/OH = 1 [working distance = 10 mm; potential = 15 kv; Magnification ×1,000 (25 μm)]. **a** SIN-P1-CO-PS0PU100, **b** SIN-P1-CO-PS10PU90, **c** SIN-P1-CO-PS20PU80, **d** SIN-P1-CO-PS30PU70 and **e** SIN-P1-CO-PS40PU60

inversion may occur. Initially (high concentration of polyurethane) the polyurethane is the continuous phase (90 and 80%). At about 75% polyurethane, a phase inversion occurs (Increasing PS amount increases the heterogeneity of the IPN structure) [2].

Conclusions

A maximum in mechanical properties was observed at around 20% of polystyrene and was explained by the increase of the crosslink density due to permanent chain entanglements. At concentrations above 20% PS content, phase separation occurred. The electron microscopy images agreed well with the glass transition behavior. Compositions [90%PU/10%PS] that are rich in PU show a continuous matrix with dispersed PS. Intermediate compositions [60%PU/40%PS] show signs of the existence of two continuous phases. IPNs based on polyurethane from castor oil modified and polystyrene show limited compatibility at low concentrations of PS, but are totally incompatible at higher concentrations of PS.

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