Scientific Paper

Semi-Interpenetrating Polymer Networks with Varying Mass Ratios of Functional Urethane and Methacrylate Prepolymers†

Alojz Anžlovar* and Majda Žigon

National Institute of Chemistry, Hajdrihova 19, SI-1000, Ljubljana, Slovenia, E-mail: alojz.anzlovar@ki.si

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† Dedicated to the memory of Prof. Dr. Tatjana Malavašič

Abstract

The morphology and mechanical properties of grafted semi-interpenetrating polymer networks (SIPNs), based on ester-urethane (PU) prepolymers and methacrylate (PM) prepolymers were studied with regard to the PU and PM mass ratio and with regard to the concentration of functional groups. The SEM micrographs, glass transition temperature shifts of the individual constituents and mechanical properties of the studied SIPNs show that their miscibility is enhanced by the interaction of complementary functional groups which also stabilize the mixture at elevated temperatures. However, enhanced miscibility was observed only in SIPNs with PU component in excess, while in SIPNs with the PM component in excess miscibility was poor. Enhanced miscibility in the SIPN with functional groups having a PU/PM mass ratio of 0.5/0.5 was ascribed to the most intense interaction between functional groups at this PU/PM ratio. Mechanical properties (Young's modulus and tensile strength) of SIPN films were defined by the polymer component forming the matrix. They were significantly influenced by the interaction between functional groups.

Key words: semi-interpenetrating polymer networks, functionalization of polymers, morphology, mechanical properties, DSC, electron microscopy

Introduction

Interpenetrating polymer networks (IPNs), composed of two or more different crosslinked polymers, are relatively new engineering materials. They have been among the fastest growing areas in the field of blends during the past twenty years.[1] Interpenetrating polymerization represents an innovative approach to solving the problem of polymer incompatibility. IPNs are defined as blends of two or more polymer networks where at least one polymer component is prepared or crosslinked in the immediate presence of others.[2–4] Besides ideal or full IPNs, in which both components are crosslinked independently of each other, there are also other types of IPNs such as semi-IPNs or grafted-IPNs. Semi-IPNs have only one component cross-linked, while grafted-IPNs have covalent cross-links between both networks.

There are a variety of combinations possible with these materials resulting in a broad range of properties. The performance of IPNs is determined by the physical and chemical nature of the constitutive networks, the relative proportion of components, the physical interactions between constitutive networks, the IPN preparation procedure, and so on.[5,6]

When IPNs are used as materials for coating applications they cannot be prepared by conventional routes (sequential and simultaneous interpenetrating polymerization) due to the presence of volatile monomers. To avoid this problem IPNs are prepared from preformed prepolymers containing complementary functional groups that enhance their miscibility.[7–12]

In order to enhance the miscibility of urethane (PU) and methacrylate (PM) functional prepolymers we incorporated tertiary amine functional groups into the PM prepolymers and carboxylic functional groups into the hard segments of the PU prepolymers In our previous work we used PU prepolymers with polyether soft segments,[13,14] while later on PU with polyester soft segments were used. In the recent publication[15] we reported on the morphology and mechanical properties of SIPNs based on ester PU and PM prepolymers with the mass ratio of 0.5/0.5 and their dependence on the concentration of functional groups. The best compatibility of polymer components in blends is in many cases found at mass ratios different to the equal mass ratio of components therefore the present work focuses on the morphology and mechanical properties of the same type of SIPNs and their dependence on the mass ratio of PU and PM functional prepolymers containing either no functional groups or 0.45 mmol of functional groups per g of polymer.

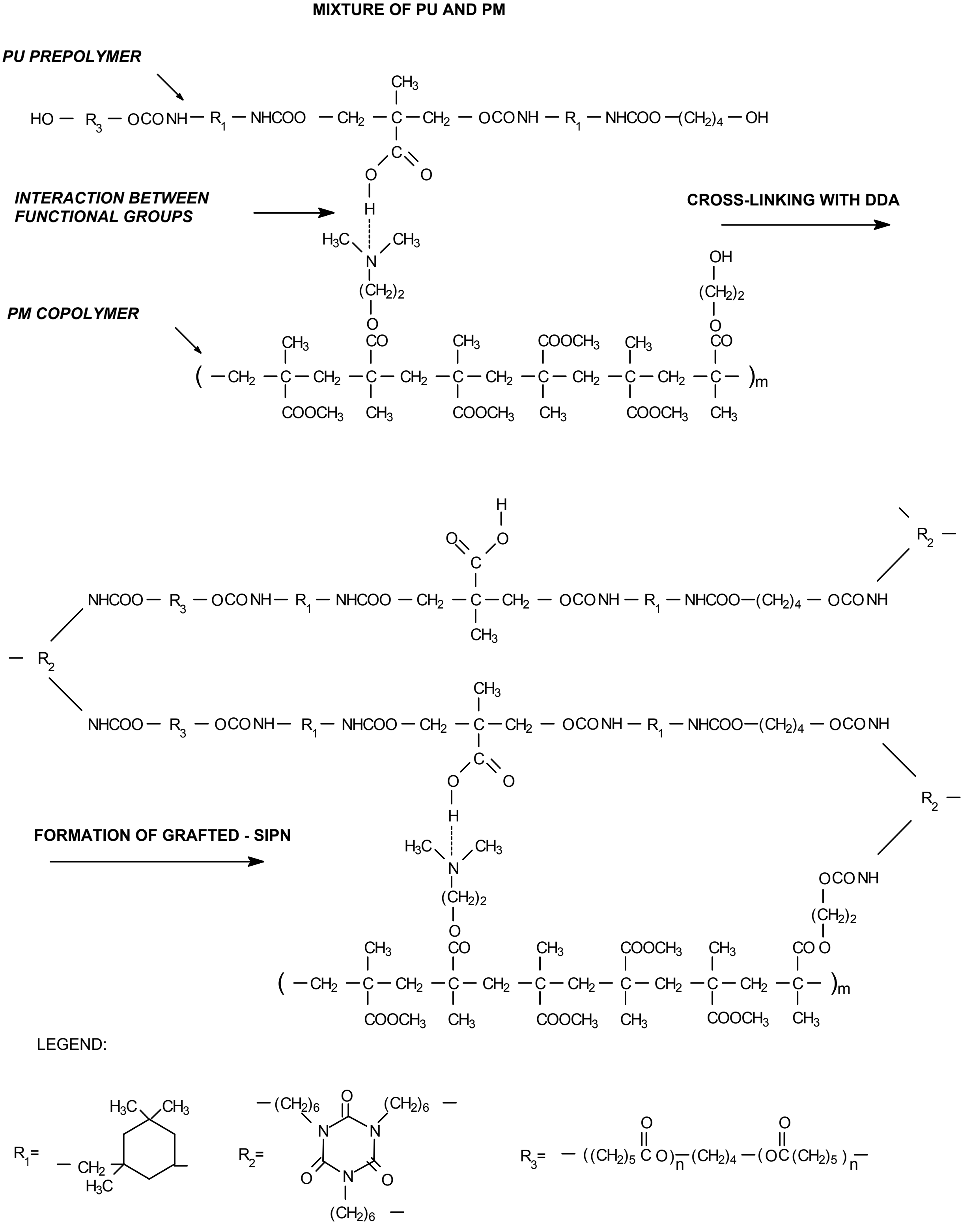

Figure 1. The scheme of preparation of SIPNs from polyester PU and PM prepolymers with functional groups.

Table 1. The number-average molecular weights - M_n of PU and PM prepolymers as measured by SEC.

PU prepolymer	Conc. of funct. groups (mmol/g)	M_n (g/mol)	PM prepolymer	Conc. of funct. groups (mmol/g)	M_n
PUp-0	0.00	12300	PMp-0	0.00	11200
PUp-45	0.45	5300	PMp-45	0.45	10700

Experimental

Synthesis of prepolymers

PU prepolymers without any, and with 0.45 mmol of carboxylic functional groups per g of polymer (PUp-0 and PUp-45) were synthesized according to prepolymer procedure described by Tirpak et al.[16] using isophoronediisocyanate (IPDI), polycaprolactone (PCL), M_n =2000, and 1,4-butanediol (BD) as monomers, and dibutyl tin dilaurate (DBTDL) as the catalyst. 2,2'-Bis-(hydroxymethyl) propionic acid (DMPA) was used as a functional comonomer to introduce carboxylic functional groups into the PUp-45 prepolymer. The molar ratio of OH/NCO groups was 1.05/1.

PM prepolymers with zero and 0.45 mmol of tertiary amine functional groups per g of polymer were synthesized by radical chain polymerization in solution (ethyl methyl ketone - MEK, reaction temperature =80 °C) from methyl methacrylate (MMA) and hydroxyethyl methacrylate (HEMA)[13,15] (0.038 mmol/g of polymer). *N*,*N*-dimethylaminoethyl methacrylate (DMAEMA) was used as a functional comonomer to introduce tertiary amine groups into the PMp-45 prepolymer. AIBN was used as an initiator and dodecylmercaptane as a regulator of the molecular weight. The PM prepolymer contains a small concentration of OH groups, therefore some covalent bonds are likely to be formed between PU and PM components. In the studied system the concentration of OH groups is constant, consequently grafting could not be the cause of the observed changes in thermal and mechanical properties of the samples studied. For simplification purposes, the term "SIPN" will be used herein to refer to grafted-SIPNs. The samples of PU and PM prepolymer were designated as PUp - X or PMp - X, where X is the concentration of functional groups in 10^{-5} mol/g of polymer.

Preparation of SIPNs

SIPNs in the form of thin films (thickness was approximately 50 μm) were prepared from prepolymer solutions in MEK. The prepolymers were mixed in a glass vessel under a constant flow of dry nitrogen in a given mass ratio of PU and PM prepolymer. The cross-linking agent [1,3,5-isocyanatohexamethylene triisocyanate, Desmodur-DA, (DDA)] was admixed in a 100% excess to the calculated amounts of OH groups in both prepolymers. The films were cast at 60 °C or at 110 °C, then kept at 60 °C for 2h and finally at 85 °C for 16h. The solvent evaporated within a few minutes at 60 °C, and in less than a minute at 110 °C. The SIPN samples were designated as IPN - X/Y, where X is the concentration of functional groups in the PU and PM prepolymers (10^{-5} mol/g of polymer) and Y is the mass fraction of the PM component. Two series of SIPN samples with functional group concentrations of 0 and 0.45 mmol/g (designation IPN - 0 and IPN – 45) were prepared. The chemical structures of prepolymers and SIPNs studied are given in Figure 1.

Methods

Size Exclusion Chromatography (SEC)

The molecular weight averages of PM and PU prepolymers were determined by SEC at 25 °C on a Perkin-Elmer liquid chromatograph consisting of a LC-250 pump, a LC-30 differential refractometer and a PL-gel Mixed D (5μm) column with a precolumn. THF was used as the eluent (flow rate 1mL/min). The columns were calibrated using PMMA standards for PM prepolymers and using PS standards for PU prepolymers. The results of the analysis are given in Table 1.

Differential Scanning Calorimetry (DSC)

The glass transition temperatures (T_g) and changes in heat capacities (Δc_p) during glass transition were determined in the temperature range from –80 °C to 130 °C. Samples were dried prior to this at 50 °C in a vacuum overnight. The measurements were performed on a Perkin-Elmer DSC-7 with a heating rate of 20 °C/min after holding the samples at –80 °C for 10 minutes. Glass transition temperatures of pure PU and PM prepolymers and the corresponding SIPNs were determined from thermograms, and their shifts in SIPNs were calculated. Conclusions about the miscibility and morphology were made on the basis of T_g shifts in SIPNs.

The Karasz-Couchman[17–19] equation (1) was used for calculation of the theoretical values of glass transitions of SIPNs, assuming an ideal miscibility of PU and PM prepolymers:

$$\ln T_{gc} = \frac{\omega_1 \Delta c_{p1} \ln T_{g1} + \omega_2 \Delta c_{p2} \ln T_{g2}}{\omega_1 \Delta c_{p1} + \omega_2 \Delta c_{p2}} \quad (1)$$

where ω_1 and ω_2 are the mass fractions of components, T_{g1} and T_{g2} are their glass transition temperatures, Δc_{p1} and Δc_{p2} are the heat capacity differences for components 1 and 2 during glass transition, respectively, and T_{gc} is the glass transition temperature of the mixture.

Mechanical Properties

Mechanical properties (tensile strength, elongation at break, Young's modulus) were measured according to the ASTM D 882-75b standard using an Instron 1022 dynamometer at a speed of 1 mm/min and with an initial grip separation of 100 mm. Hardness of thin films was determined by Koenig's method (DIN 53157).

Scanning Electron Microscopy (SEM)

Samples broken on the dynamometer were coated twice with gold, and micrographs of the fractured surfaces were taken using a JEOL JSM 840 A microscope operating at an acceleration voltage of 15 keV (magnification 1000x) in secondary electron emission mode.

Results and Discussion

SEM micrographs of the IPN-0 and IPN-45 series with different mass ratios of PU and PM components are shown in Figure 2. The phase separation is much more pronounced in SIPNs with no functional

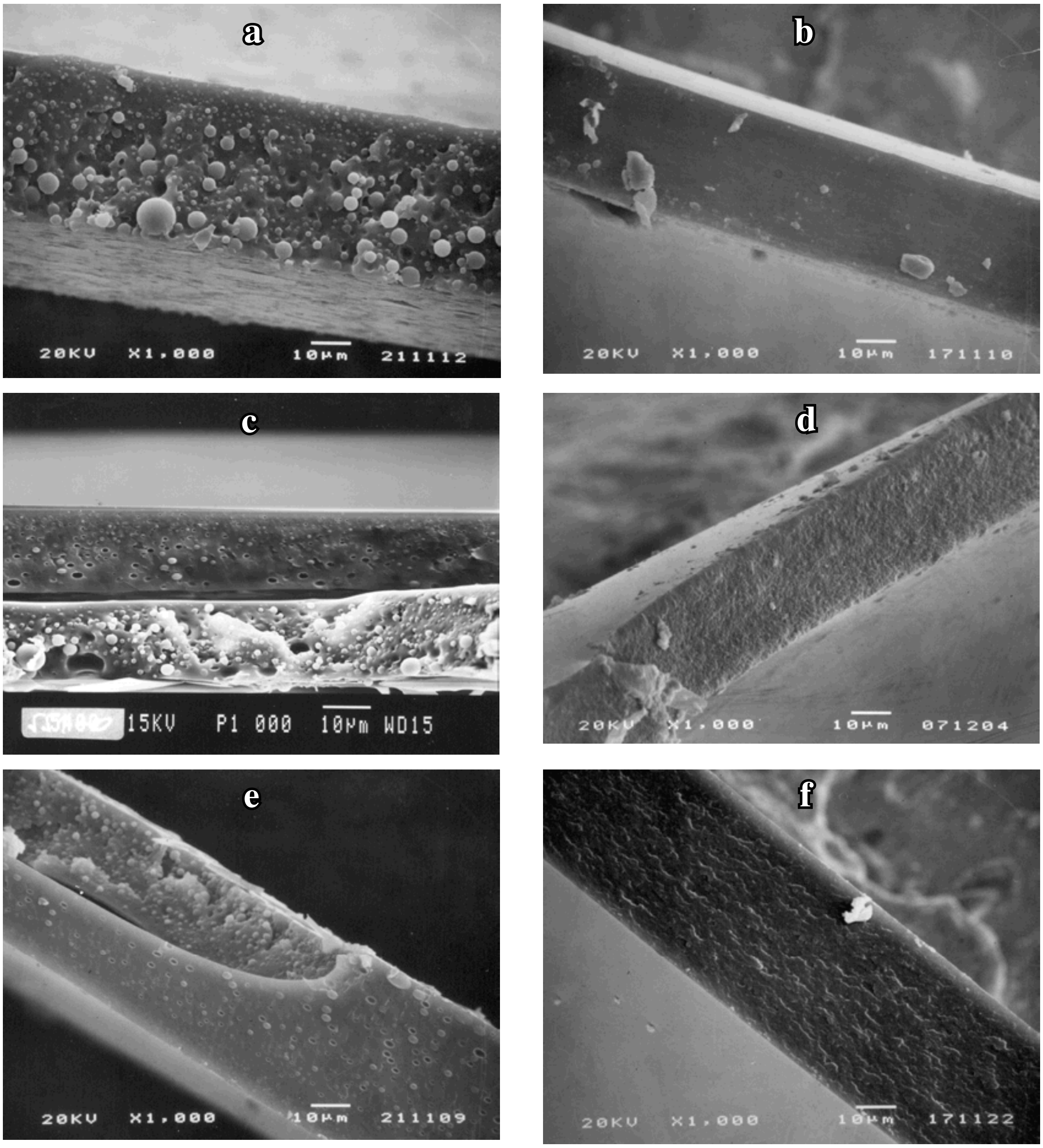

Figure 2. SEM micrographs (magnif. - 1000x) of IPNs cast at 60 °C with no functional groups (IPN 0, PU/PM = a) 0.7/0.3, c) 0.5/0.5, e) 0.3/0.7) and with 0.45 mmol/g of functional groups (IPN-45, PU/PM = b) 0.7/0.3, d) 0.5/0.5, f) 0.3/0.7).

groups (IPN-0, Figure 2 a,c,e) than in SIPNs with functional groups (IPN-45, Figure 2 b,d,f), which show relatively homogenous morphologies. Comparing the micrographs of IPN-45 series with different PU/PM mass ratios it can be observed that phase separation is reduced, if one component is in excess, especially when it is the PU component that is in large excess (Figure 2b). Differences in morphologies lead to the conclusions that functional groups substantially improve the compatibility of PU and PM prepolymers and that SIPNs with very homogeneous morphologies can be obtained, when the PU component is in excess.

Glass transition temperatures as a function of the PU and PM prepolymer mass ratio of the IPN-0 and IPN-45 series cast at 60 °C are shown in Figure 3. Comparison of the T_g values of PU soft segments and of PM component for IPN-45 and for IPN-0 samples with theoretical values calculated using Karasz-Couchman equation shows that SIPN samples of both series can be divided into three groups.

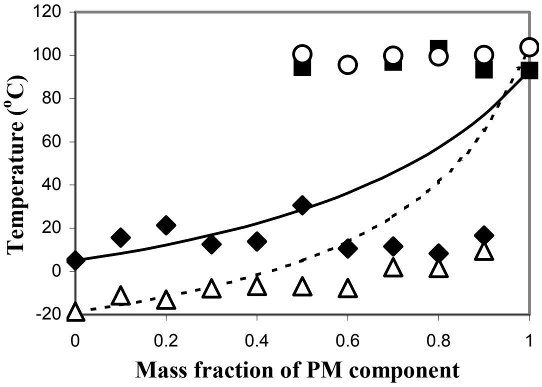

Figure 3. Glass transition temperatures of PU soft segments and PM component in IPN samples with no functional groups (IPN 0) and with 0.45 mmol/g of functional groups (IPN 45) cast at 60 °C in dependence of the PU/PM mass ratio: **IPN 0** - (PU soft s. -(Δ), PM comp. -(○)); **IPN 45** - (PU soft s. -(◆), PM comp. -(■)); calculated values by the equation 1: IPN 0 (---), IPN 45 (—).

The first group are SIPN samples with the PU component in excess. In these samples, where PU forms the matrix, we observe shifts in the T_g values of the PU soft segments towards higher temperatures. Shifts are observed in the IPN-45 and IPN-0 series, and T_g values are close to the values calculated using the Karasz-Couchman equation, thus indicating enhanced miscibility of the PU and PM component in both series, although enhancement is more pronounced in the IPN-45 series. Comparing these T_g shifts with those of the polyether PU soft segments in SIPNs, it is evident that PU prepolymers with polyester soft segments are more compatible with PM prepolymer than with polyether ones.[13] Better compatibility of polyester PU soft segments in the SIPNs is caused by the increased intensity of interaction (increased number of hydrogen bonds) due to the presence of a polar ester carbonyl group.[20,2] The glass transitions of the PM component could not be observed in these SIPN samples. Our explanation for this phenomenon is that glass transition temperatures are shifted to lower temperatures and are hidden in intense glass transitions of the PU soft segments.

The second group are SIPN samples with the PM component in excess. The glass transition temperatures of the PM component were observed in these samples but they shifted to slightly lower or higher temperatures compared to the pure PM. Most of these changes are within the range of experimental error indicating that at least a part of the PM component is not interpenetrated with the PU component. Glass transition temperatures of PU soft segments are 20 to 40 °C lower than the values calculated using the Karasz-Couchman equation. This indicates the poor miscibility of components in SIPNs where the PM component forms a matrix.

The third group are IPN-0 and IPN-45 samples with equal mass ratios of PU and PM prepolymers. Here we observe different shifts in T_g of PU soft segments in IPN-0 and in IPN-45 samples compared to the calculated values. The glass transition temperature of the IPN-0/0.5 is 12 °C lower than the calculated value while the measured value of the IPN-45/0.5 is close to the calculated one, thus indicating enhanced miscibility. Glass transition temperature shifts of the PM component are shifted slightly towards higher or lower temperatures compared to T_g values of the pure PM. These shifts are in the range of experimental error and it can be therefore inferred that a part of the PM component is not interpenetrated with PU chains. This was observed also at other IPN systems.[6]

Shifts in the T_g of PU soft segments clearly indicate enhanced miscibility in the IPN-45/0.5 sample and poor miscibility in the IPN-0/0.5 sample, thereby confirming that functional groups do enhance the miscibility of the components. The compatibilizing effect of functional groups is most highly expressed at an equal mass ratio of PU and PM components because functional groups are in an equimolar ratio and therefore interaction between functional groups is the most intense at this mass ratio of PU and PM component.

Films of SIPN samples were prepared also by casting the MEK solution at 110 °C, which substantially reduces the solvent evaporation time. The main difference between the glass transition temperatures of PU soft segments of the IPN-0 and IPN-45 samples cast at both temperatures is shown in Figure 4. While T_g values of PU soft segments of the IPN-45 samples do not show any important differences, there is a major difference in T_g shifts of PU soft segments between IPN-0 samples cast at 60 and those at 110 °C. T_g values of the IPN-0 samples cast at 60 °C shift to higher temperatures with the increased mass ratio of the PM component, while the T_g values in IPN-0 samples cast at 110 °C are practically independent of the mass ratio of components thus indicating their phase separation and poor miscibility of the components. Phase separation is most probably the consequence of the reduced viscosity of the mixture at elevated temperatures. In the IPN-45 series the mixture is stabilized by the interaction between the functional groups[22] and in these samples phase separation is substantially reduced. A similar conclusion was also drawn from the polarizing micrography and WAXD data. It was found in the IPN-45/0.5 that this interaction prevented ordering of domains in the PU component.[23]

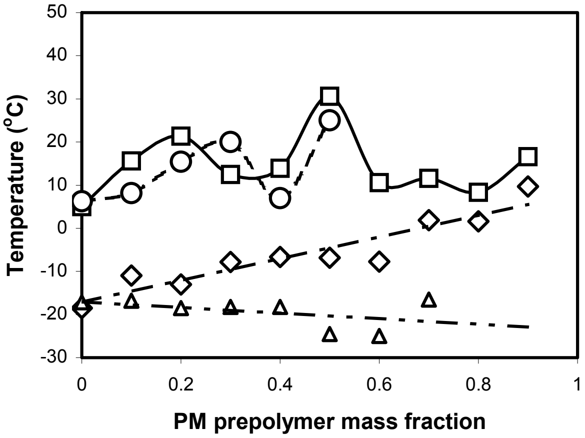

Figure 4. Glass transition temperatures of PU soft segments in IPN samples dependent on the mass ratio of the PU and PM component: IPN-0 series cast at 60 °C (◇) and at 110 °C (∆), IPN-45 series cast at 60 °C (□) and at 110 °C (○).

In general, glass transition temperatures of the PM component seem to be independent of the concentration of functional groups and the mass ratio of both components, while T_g shifts of PU soft segments indicate enhanced miscibility of components, if the PU component forms a matrix or if the mass ratios of both components are equal. The fact that T_g values of the PM component remain practically the same does not necessarily mean that the miscibility of the components in the SIPN samples is poor. Namely, ESR studies of the IPN-45/0.5 sample confirmed that PM chains are partially plasticised by the PU component,[23] indicating an enhanced miscibility in this sample. The observed anomaly in glass transition temperatures of the PM component can be ascribed to:

- the compensation of the plasticising effect of the PU component by the interaction between functional groups acting as physical cross-linking.[24] Chemical cross-linking may be an additional contribution to this. Physical and chemical crosslinking shifts the temperature of the glass transition towards higher temperatures,[25–29]
- the structure of the PM prepolymer – tertiary amine functional groups are built into side chains creating steric hindrance and preventing the PM main chain from coming into close contact with the main chain of the PU prepolymer,[30]
- non-random distribution of functional groups along polymer chains of prepolymers due to the segmented structure of the PU prepolymer and due to nonideal copolymerisation reaction of the PM prepolymer.[15]

The mechanical properties of SIPNs with different mass ratios of the PU/PM prepolymer without (IPN-0 series) and with functional groups (IPN-45 series) are given in Table 2 and Figure 5. Only SIPN films cast at 60 °C were suitable for testing mechanical properties, since those cast at 110 °C contained bubbles formed by the fast evaporation of the solvent.

Mechanical properties of SIPN films were primarily defined by the polymer component in excess that forms the matrix. This is seen from the substantial drop of the elongation and from the substantial increase of the Young's modulus in the region of the equal mass ratios of components in both series where phase inversion takes place[31] (Table 2, Figure 5). Nevertheless, the same results show some important differences between IPN-0 and IPN-45 series. The tensile strength of the IPN-0 samples is lower than that of the pure PU prepolymer (PUp-0), while the tensile strength of the IPN-45 samples increases up to a PU/PM mass ratio 0.5/0.5 (Table 2) and then rapidly deteriorates. The Young's moduli of SIPNs without functional groups are much lower than those of SIPNs with functional groups. In the IPN - 0 series the dependence of Young's modulus on the mass fraction of the PM component is sigmoidal, indicating phase inversion in the steep slope of the curve (PU/PM mass ratio = 0.5/0.5), while in the IPN-45 series the modulus starts to increase at a PU/PM mass ratio of 0.7/0.3 and has a maximum at a ratio of 0.4/0.6 indicating that strong interactions are present at these PU/PM ratios (Figure 5). It can be concluded from these results that, by adding between 40 and 50 mass % of the PM prepolymer to the PU component, the Young's modulus can be substantially increased (Figure 5). However, 30 mass % or less PM does not so greatly change any of the mechanical properties (except Koenig's hardness) of the SIPNs, when compared to the pure PU prepolymer. This can be important from the technological point of view because polyacrylates are much cheaper than polyurethanes.

Koenig's hardness as a function of composition (Figure 5) also differs between the two series. The IPN-0 series has a typical sigmoidal shape, while the curve of the IPN-45 series starts to increase substantially from the pure PU component, reaches a maximum at PU/PM

Table 2. Tensile strength and elongation of films of IPN - 0 and IPN - 45 series with different PU/PM mass share ratios; films cast at 60 °C.

PU/PM mass ratio in IPN-0	Tensile strength (N/mm^2)	Elongation (%)	PU/PM mass ratio in IPN-45	Tensile strength (N/mm^2)	Elongation (%)
1.0/0.0	12.5 ± 1.4	492 ± 15	1.0/0.0	20.0 ± 2.10	380 ± 20
0.9/0.1	3.50 ± 1.2	350 ± 73	0.9/0.1	13.3 ± 0.84	358 ± 13
0.8/0.2	3.37 ± 0.57	332 ± 61	0.8/0.2	17.3 ± 1.53	347 ± 24
0.7/0.3	7.10 ± 2.0	365 ± 63	0.7/0.3	20.4 ± 1.36	302 ± 20
0.6/0.4	3.20 ± 0.32	202 ± 20	0.6/0.4	21.7 ± 1.10	248 ± 11
0.5/0.5	2.78 ± 0.52	37.5 ± 19	0.5/0.5	24.0 ± 1.05	86.7 ± 27
0.4/0.6	2.18 ± 0.25	5.9 ± 2.8	0.4/0.6	9.74 ± 5.2	1.6 ± 0.51
0.3/0.7	1.83 ± 0.26	2.3 ± 0.55	0.3/0.7	2.15 ± 0.9	0.79 ± 0.24
0.2/0.8	–	–	0.2/0.8	–	–
0.1/0.9	–	–	0.1/0.9	–	–

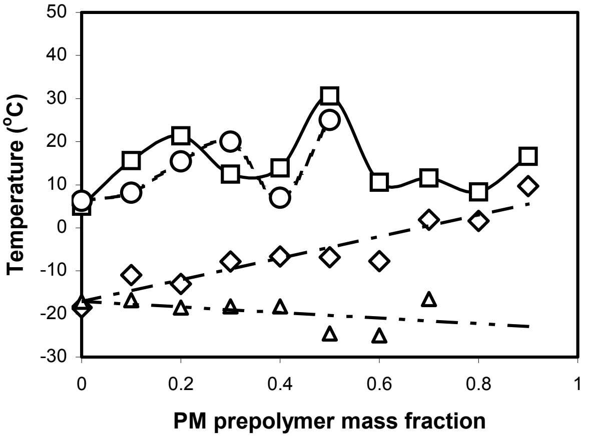

Figure 5. Young's modulus of IPN - 0 (○) and IPN - 45 series (Δ) and Koenig's hardness of IPN - 0 (∇) and IPN - 45 series (□) as a function of the PM prepolymer mass fraction.

mass ratio of 0.4/0.6 and then slightly decreases as the composition goes towards the pure PM component. This is the most pronounced difference in miscibility of PU and PM components between the IPN-0 and IPN-45 series.

Conclusions

A study was made on the morphology and mechanical properties of SIPNs with different PU/PM mass ratios without (IPN-0) and with complementary functional groups at a concentration of 0.45 mmol/g (IPN-45) based on ester-urethane (PU) prepolymers with carboxylic groups and methacrylic (PM) prepolymers with tertiary amine functional groups.

SEM micrographs and shifts in T_g showed enhancement of miscibility of PU and PM components in IPNs with high concentration of functional groups. According to the shifts in T_g and SEM micrographs of the IPN samples with differrent PU/PM mass ratios miscibility of components is enhanced in IPNs in which PU component forms the matrix while no enhancement was observed in IPNs in which matrix is formed by the PM component. The sample with an equal PU/PM mass ratio having 0.45 mmol of functional groups/g of polymer shows exceptional enhancement of miscibility due to an intense interaction between complementary functional groups which are in the equimolar ratio at this PU/PM mass ratio. Interaction between functional groups also stabilizes the PU and PM prepolymer mixture at elevated temperatures (110 °C) and thus prevents the separation of components under these conditions.

Mechanical properties of SIPN films in general were defined by the polymer component in excess and forming the matrix, although interaction between functional groups also influenced their properties resulting in increased Young's modules and tensile strengths compared to those without functional groups. Mechanical properties of SIPNs (Young's modulus and Koenig's hardness) as a function of the PU/PM mass ratio supported conclusions about the enhanced compatibility of PU and PM prepolymers due to interaction between functional groups.

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Povzetek

Sintetizirali smo ester-uretanske (PU) predpolimere z vgrajenimi karboksilnimi funkcionalnimi skupinami in polimetakrilatne (PM) predpolimere z vgrajenimi terciarnimi aminskimi funkcionalnimi skupinami in iz njih pripravili delno prepletene polimerne mreže (SIPN) pri različnih temperaturah. Študirali smo morfologijo in mehanske lastnosti SIPN v odvisnosti od koncentracije funkcionalnih skupin in od razmerja med predpolimeroma. Premiki temperature steklastega prehoda in SEM mikrografije kažejo povečanje mešjivosti PU in PM komponent zaradi interakcije med funkcionalnimi skupinami. V SIPN s presežkom PU komponente je mešljivost PU in PM predpolimera izboljšana, medtem ko v vzorcih SIPN s presežkom PM komponente predpolimera nista kompatibilna. Najboljša mešljivost komponent v SIPN je pri razmerju PU/PM = 0.5/0.5, ker so interakcije pri tem razmerju (ekvimolarno razmerje funkcionalnih skupin) najbolj intenzivne. Interakcije med funkcionalnimi skupinami vplivajo tudi na mehanske lastnosti SIPN, vendar pa njihove vrednosti v največji meri določa komponenta, ki tvori matrico.