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Interpenetrating polymer networks of poly(styrene-*co*-4-vinylphenyldimethylsilanol) and poly(*N*-vinylpyrrolidone): a new approach to network formation

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Blends consisting of poly(styrene-*co*-4-vinylphenyldimethylsilanol) (ST–VPDMS) and poly(*N*-vinylpyrrolidone) (PVPr) were studied by glass transition temperature measurements and *FT*i.r. spectroscopy. It was found that ~4 mol% of VPDMS monomer units had to be incorporated into styrene copolymers in order to form miscible blends with PVPr. Mutual precipitates in the form of interpolymer complexes were obtained for the ST–VPDMS copolymers containing more than 34 mol% VPDMS. Semi-interpenetrating polymer networks (semi-IPNs) were prepared from ST–VPDMS/PVPr interpolymer complexes by self-condensation of dimethylsilanol groups when the copolymers contained more than 60 mol% VPDMS, and thus provided a new route to IPN formation without the presence of any external crosslinkers or catalysts.

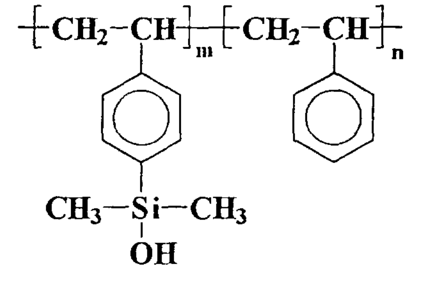
(Keywords: polymer blends; interpolymer complexes; polymer networks)

INTRODUCTION

In a recent publication[1], we reported the synthesis of the 4-vinylphenyldimethylsilanol polymer and its styrene copolymers (*Scheme 1*), which was achieved by oxygen atom insertion into the Si–H bond via reaction with dimethyldioxirane. In the course of that study, we found that the dimethylsilanol groups had a strong tendency to undergo spontaneous condensation to form siloxane crosslinkages in the solid state. The chemical stability of poly(styrene-*co*-4-vinylphenyldimethylsilanol) (ST–VPDMS) decreased with increasing amounts of silanol units in the copolymer. However, the dimethylsilanol groups in both the homopolymer and the copolymers were found to be stable in acetone solution at room temperature due to the formation of hydrogen bonds between the silanol group and the solvent.

The silanol groups are known to be strong hydrogen bond donors. However, they also self-associate strongly[1–3]. Studies on the blends of poly(styrene-*co*-4-vinylphenyldimethylsilanol) with poly(n-butyl methacrylate) (PBMA) showed that miscible blends were formed only for the copolymers containing 4 to 34 mol% VPDMS[3,4]. In these miscible blends, the formation of intermolecular hydrogen bonds between the silanol and carbonyl groups minimized the fraction of self-associated silanol groups and hence the self-condensation of the silanols. For copolymers containing more than 34 mol% VPDMS, the strong self-association of the silanols led to phase separation. The self-associated silanol groups in the immiscible blends were readily converted into siloxane crosslinkages and resulted in the formation of phase-separated, semi-interpenetrating polymer networks (semi-IPNs)[5].

The primary objective of this present investigation was to seek a suitable acceptor polymer to extend the miscibility window of the silanol-containing polymer blends, and to utilize the dual nature of the silanol groups to prepare miscible semi-IPNs by self-condensation of silanols. Poly(*N*-vinylpyrrolidone) (PVPr) was chosen for this study because the amide group was known to be a stronger hydrogen bond acceptor than the ester carbonyl group of PBMA[6–8]. Once intimate mixing of the component polymers is achieved over a wide range of copolymer compositions in the blends, we could then make use of the spontaneous condensation of the silanol groups to prepare semi-interpenetrating polymer networks from these miscible blends.



Scheme 1

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EXPERIMENTAL

Materials

Poly(*N*-vinylpyrrolidone) (PVPr), with a reported M_w of 400 000, was purchased from Aldrich Chemical Co. The polymer was dried in a vacuum oven at 80°C for 2 days and then stored in a desiccator under vacuum before use. The 4-vinylphenyldimethylsilanol homopolymer (PVPDMS) and styrene copolymers (ST–VPDMS) were synthesized from the related 4-vinylphenyldimethylsilane homopolymer and styrene copolymers, as described earlier[1]. For convenience in sample identification, the numeral following PVPDMS indicates the mole percentage of VPDMS monomer units in the copolymers (*Table 1*).

Preparation of blends, interpolymer complexes and semi-interpenetrating polymer networks (semi-IPNs)

Solutions (2% wt/vol) of poly(styrene-*co*-4-vinylphenyldimethylsilanol) copolymers[3] and poly(*N*-pyrrolidone) (PVPr) in chloroform were prepared. The blends were prepared by mixing appropriate amounts of each polymer solution while stirring. When the copolymer contained ≥34 mol% VPDMS, mutual precipitation of the two polymers took place; these precipitates were filtered and washed with chloroform and acetone, respectively, followed by vacuum drying at 80°C to constant weight. For thermal analysis, samples were prepared by casting films on glass slides, and for infra-red analysis, films were cast on potassium bromide windows. After the majority of the solvent had been slowly removed by evaporation at room temperature, the films were dried under vacuum at 80°C for 4 days under nitrogen (for protection). In the case of the mutual precipitates, KBr discs were prepared for infra-red analysis in the usual way.

Thermal analysis

Differential scanning calorimetry (d.s.c.) was performed with a Perkin-Elmer DSC-7 calorimeter, with all the data being processed by means of the Perkin-Elmer TADS system. Sample weights of 5 to ~10 mg, and a heating rate of 20°C min^{-1}, were used. The sample was first heated from 30 to 210°C and maintained at that temperature for 3 min; it was then cooled to 30°C in the d.s.c. cell and kept for 5 min. The sample was then re-scanned and the midpoint of the specific heat increment in the second scan was taken as the glass transition temperature.

Table 1 Composition and characterization of poly(styrene-*co*-4-vinylphenyldimethylsilanol) and poly(*N*-vinylpyrrolidone) (PVPr)

Sample	VPDMS content (mol%)[a]	$[\eta]^b$ (dl g^{-1})	T_g (°C)
PVPDMS-2	1.9	0.54	97
PVPDMS-4	4.1	0.58	98
PVPDMS-9	8.7	0.64	98
PVPDMS-11	11.4	0.59	99
PVPDMS-18	18.2	0.67	101
PVPDMS-34	33.9	0.70	115
PVPDMS-60	60.0	0.76	121
PVPDMS-81	80.6	0.66	135
PVPDMS-100	100	0.55	147
PVPr	-	-	178

[a] Calculated from ^{1}H n.m.r. spectra

[b] Viscosities of the precursor silane copolymers, or homopolymer, were measured in THF at 25±0.01°C

Table 2 Characteristics of the 50/50 wt/wt blends of poly(styrene-*co*-4-vinylphenyldimethylsilanol) and poly(*N*-vinylpyrrolidone)

No.	Blend code	Blend solution appearance	Film or complex appearance	T_g (°C)
1	PVPDMS-2	Clear	Opaque	99, 157
2	PVPDMS-4	Clear	Transparent	105
3	PVPDMS-9	Clear	Transparent	113
4	PVPDMS-11	Clear	Transparent	116
5	PVPDMS-18	Clear	Transparent	117
6	PVPDMS-34	Precipitation	Transparent	135
7	PVPDMS-60	Precipitation[a]	Transparent	183
8	PVPDMS-81	Precipitation[a]	Transparent	–[b]
9	PVPDMS-100	Precipitation[a]	Transparent	–[b]

[a] Insoluble in either dimethyl sulfoxide (DMSO) or dimethylformamide (DMF)

[b] No glass transition temperatures were observed for these complexes in the temperature range from 50 to 300°C

FT*i.r. spectroscopy*

Infra-red spectra were measured on a Digilab FTS-60 Fourier transform infra-red spectrometer, with a minimum of 64 scans, at a resolution of 2 cm^{-1}, being signal-averaged in this work. Films used in the *FT*i.r. measurements were sufficiently thin to be within the absorbance range where the Beer–Lambert law was obeyed.

RESULTS AND DISCUSSION

The compositions and characteristics of the ST–VPDMS copolymers and PVPr used in the blending studies are shown in *Table 1*. In the ST–VPDMS copolymers, the dimethylsilanol groups can act as strong hydrogen bond donors, but they also have a tendency to undergo spontaneous condensation to form siloxane crosslinks in the solid state when the silanol groups are present at levels of 18 mol% or higher[1].

General observation on the miscibility of the ST-VPDMS/PVPr blends

The results obtained for 50/50 wt/wt ST–VPDMS/PVPr blends are shown in *Table 2*. The mixed solutions of the two polymers were clear when the ST–VPDMS copolymers contain less than 34 mol% VPDMS units. When the VPDMS content in the copolymer is 34 mol% or higher, the strong interaction between the polymers resulted in the formation of interpolymer complexes in the form of precipitates. In most instances, a single T_g was obtained for the precipitates; in a few cases, the T_g escaped detection up to 300°C. An opaque film was observed for the blends of 50/50 wt/wt PVPDMS-2 with PVPr. Glass transition temperature measurements indicated that this blend of PVPDMS-2 with PVPr was heterogeneous and showed two distinct glass transition temperatures. Miscible blends were formed for PVPr with PVPDMS-4, PVPDMS-9, PVPDMS-11 and PVPDMS-18, as defined by the presence of a single glass transition temperature.

The composition dependence of the glass transition temperatures of the miscible PVPDMS-18/PVPr blends were measured for the entire composition range and the

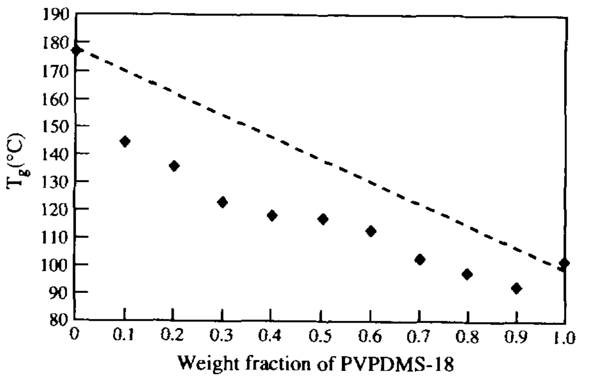

Figure 1 Composition dependence of the glass transition temperatures of PVPDMS-18/PVPr blends

results are shown in *Figure 1*. In contrast to some hydrogen bonded polymer blends where the positive deviations of glass transition temperatures from the calculated weight-average values were observed[9–12], the glass transition temperatures of the PVPDMS-18/PVPr blends were found to fall below the calculated weight-average values. In addition, the T_g values of the blends containing 80 and 90 wt% PVPDMS-18 even fell below the value of the lower-T_g-component polymer (PVPDMS-18). In these experiments, the sample was dried at 80°C for 4 days and the T_g was taken from the second scan after the sample had been heated to 210°C and maintained at that temperature for 3 min. The possibility of plasticization of the blends by residual chloroform solvent could be eliminated. We speculate that the low T_g values of the PVPDMS-18/PVPr blends may be attributed to the less extensive hydrogen bonding interactions between PVPDMS-18 and PVPr, which results in poor packing of the chain segments, and therefore a large free volume increment in the blends. We also found that correlation of the T_gs with blend composition cannot be fitted by using conventional equations[10,13–15].

FT*i.r. studies of the ST–VPDMS/PVPr blends*

*Silanol-stretching-vibration region of poly(styrene-*co*-4-vinylphenyldimethylsilanol).* *Figure 2* shows the hydroxyl-stretching-vibration region of the infra-red spectra of poly(styrene-*co*-4-vinylphenyldimethylsilanol) (ST–VPDMS) containing varying amounts of 4-vinylphenyldimethylsilanol (VPDMS)[1]. There were two absorption bands in the silanol-stretching-vibration region of the infra-red spectra. The band at 3622 cm^{-1} could be attributed to the free silanol stretching vibration and the broad bands ranging from 3435 to 3319 cm^{-1} were assigned to the broad distribution of the self-associated silanols[16]. The infra-red absorption band of the self-associated silanol groups was characterized by a substantial width and a large integral intensity relative to the absorption band of the free silanol groups. With increasing VPDMS content in the copolymer, the absorbance of the self-associated silanol bands increased at the expense of that of the free silanol bands, and the location of the maximum of the self-association band shifted to a lower wavenumber. The frequency shifts ($\Delta\nu$) caused by the self-association were in the range from 187 to ~303 cm^{-1} (*Figure 3*) and depended on the amount of VPDMS in the copolymers. The magnitude of the frequency shifts, and hence the strength of the self-association, increased dramatically when the amount of VPDMS in the copolymer was in excess of 10 mol%.

Silanol-stretching-vibration region of PVPDMS-18/PVPr blends. The infra-red spectrum of PVPDMS-18 (*Figure 4*, E) in the silanol stretching region was characterized by a strong broad band centred at 3365 cm^{-1} (self-associated) and a relatively weak band at 3622 cm^{-1} (free), as discussed before. For the blends, the free silanol absorption band at 3622 cm^{-1} was diminished as the amount of PVPr increased and eventually disappeared for blends containing more than 50 wt% PVPr. A new band centred at 3345 cm^{-1}, assigned to the silanol group which was hydrogen bonded to the amide carbonyl group, appeared. The absorption band of the self-associated silanol groups was observed at relatively higher wavenumbers (3416 cm^{-1}) and this slightly shifted to lower wavenumbers with increasing PVPDMS-18 content in the blends. The location of the

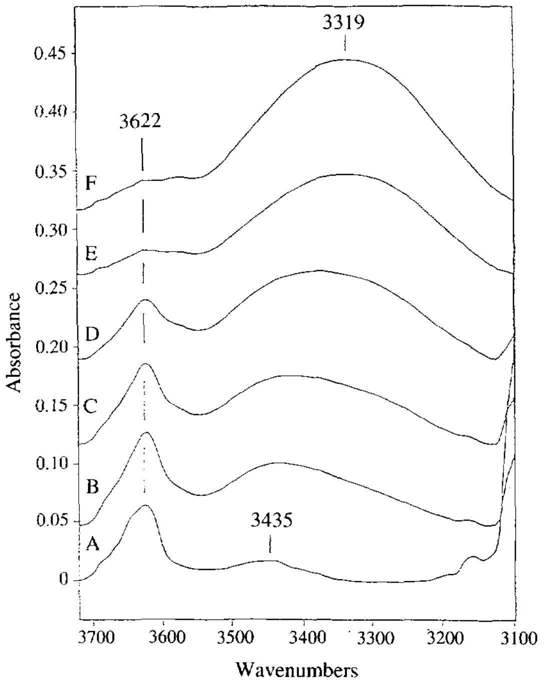

Figure 2 FT*i.r.* spectra in the hydroxyl-stretching-vibration region for poly(styrene-*co*-4-vinylphenyldimethylsilanol) containing: (A) 2; (B) 9; (C) 11; (D) 18; (E) 60; (F) 100 mol% VPDMS

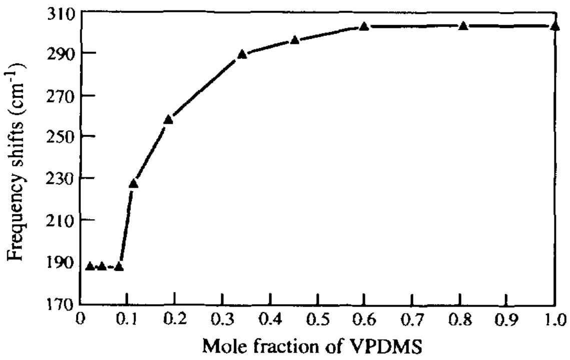

Figure 3 Plots of the frequency shifts as a function of the VPDMS contents in the styrene copolymers

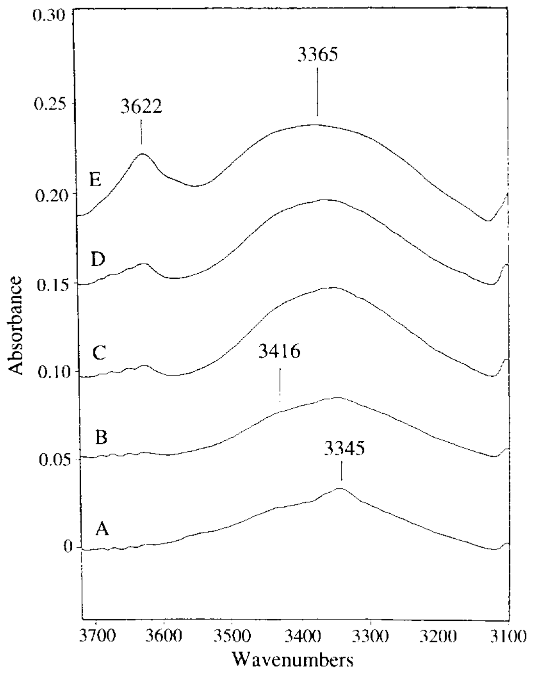

Figure 4 Infra-red spectra in the silanol-stretching-vibration region for (A) 30/70, (B) 50/50, (C) 70/30, and (D) 80/20 PVPDMS-18/PVPr blends, and (E) PVPDMS-18

peak maximum of the PVPDMS-18/PVPr blends shifted gradually to higher wavenumbers as the content of PVPDMS-18 increased in the blends. The frequency shift ($\Delta\nu$) between the free and hydrogen bonded silanol absorption bands for PVPDMS-18/PVPr was 277 cm^{-1}, which was larger than the value observed for the self-associated silanols (257 cm^{-1}) in the PVPDMS-18 copolymer. The large frequency shifts observed for hydrogen bonded silanols suggest that a strong intermolecular hydrogen bond interaction occurs in the ST–VPDMS/PVPr blends. In sharp contrast to the above, the frequency shift caused by the intermolecular hydrogen bond between the dimethylsilanol group of the ST–VPDMS and the carbonyl group of PBMA[3,4] was much smaller, i.e. only ~94 cm^{-1}.

Amide-carbonyl-stretching-vibration region of PVPDMS-18/PVPr blends. The amide-carbonyl-stretching-vibration band of PVPr was located at 1682 cm^{-1} (*Figure 5*). A new strong absorption band at 1663 cm^{-1} appeared upon blending PVPDMS-18 with PVPr, which was assigned to the hydrogen bonded amide carbonyl band[6]. The absorbance of this band increased with increasing amounts of PVPDMS-18 in the blends. The spectral features of the ST–VPDMS/PVPr blends in the amide-carbonyl-stretching region are also in sharp contrast with that observed for the corresponding PBMA blends[3,4] in the carbonyl-stretching region; in the latter, only a weak shoulder was observed at 1703 cm^{-1}.

To obtain quantitative information concerning the intermolecular hydrogen bond interactions in the amide-carbonyl-stretching-vibration region of the PVPDMS-18/PVPr blends, a curve-fitting program was used to deconvolute the overlapped bands in the amide-carbonyl-stretching region. A representative curve-resolved spectrum is shown in *Figure 6*. In the curve-fitting process, a flat baseline was set between 1730 and 1620 cm^{-1}.

The composite spectra profiles in the amide-carbonyl-stretching-vibration region were found to be satisfactorily fitted by a Voigt profile, with two component bands centred at ~1684 and 1660 cm^{-1}, respectively. We have noted that the fraction of the total peak area due to the hydrogen bonded amide carbonyl group increased with increasing content of PVPDMS-18 in the blends (*Figure 7*). Since the band observed at 1682 cm^{-1} for PVPr is a mixed mode containing contributions from both the carbonyl and N–C stretching vibrations[6], we could not directly correlate the areas of the two bands with the amounts of the hydrogen bonded and non-hydrogen bonded amide carbonyl groups in the blends, as in the case of the ester carbonyl group[3]. However, the results obtained from the curve-fitting procedure clearly indicate the overall trend for the fraction of hydrogen bonded amide carbonyl groups in these blends.

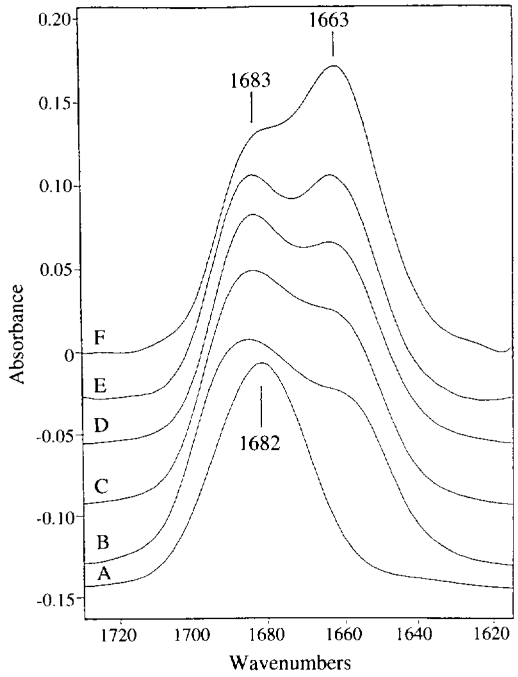

Figure 5 Infra-red spectra in the amide-carbonyl-stretching vibration region for (A) pure PVPr, and (B) 50/50, (C) 60/40, (D) 70/30, (E) 80/20 and (F) 90/10 PVPDMS-18/PVPr blends

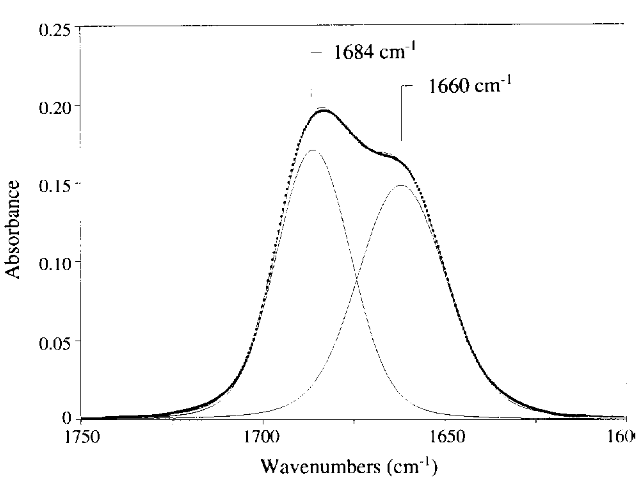

Figure 6 Curve-resolved spectrum of the 60/40 PVPDMS-18/PVPr blend in the amide-carbonyl-stretching-vibration region

Semi-interpenetrating polymer networks of the ST–VPDMS/PVPr system

In contrast to the results reported previously for the corresponding PBMA blends[3,4] there was no upper limit for the miscibility window of the 50/50 wt/wt ST–VPDMS/PVPr blends. Instead, mutual precipitates were formed from solutions when the copolymer contained $\geqslant$34 mol% VPDMS (*Table 1*). The yield and compositions of the complexes are listed in *Table 3*. As seen in this table, the yields of the precipitated interpolymer complexes, as might be expected, increase with increasing VPDMS content of the copolymers, due to the increased extent of interpolymer hydrogen bonding interactions. We have also noted that the molar ratios of the Si–OH/C=O groups in the complexes decrease slightly with increasing VPDMS content of the copolymers, which indicate that there is more PVPr hydrogen bonded to the copolymer in the precipitated complexes as a direct result of increased hydrogen bonding interactions between the two polymers.

In preparing the complexes, we have noted that they became insoluble in dimethyl sulfoxide (DMSO) or dimethylformamide (DMF) after they were isolated from the solutions, with the exception of the PVPDMS-34/PVPr complex. Tough, transparent materials were produced after drying at 80°C for 4 days. The glass transition temperature of the PVPDMS-34 complex (135°C) was still much lower than that of the higher-T_g-component polymer (T_g of PVPr is ~178°C), as well as the weight-averaged value (146°C). However, the glass transition temperature of the PVPDMS-60 complex was found to be 183°C, higher than the values of both component polymers and the PVPDMS-34 complex (*Table 1*). The higher T_g value could be attributed to an extensive strong intermolecular hydrogen bonding interaction between the silanol and amide carbonyl groups, plus possibly the formation of siloxane crosslinkages by condensation of the silanols.

The infra-red spectra of the complexes are shown in *Figure 8*. In the silanol-stretching-vibration region, the silanol-stretching-vibration band was characterized by a substantially broad band centred at ~3398 cm^{-1}. This broad band consisted of the contributions from the stretching vibrations of both the amide-bonded silanol and the self-associated silanol (almost all of the free silanols were readily bonded to the amide-carbonyl groups). In the amide-carbonyl-stretching-vibration region, the amide-carbonyl-stretching-vibration band was split into two bands, as was the case for that discussed above for the miscible PVPDMS-18/PVPr blends.

In previous studies, we have found that the dimethylsilanol groups had a strong tendency to undergo spontaneous condensation and transform into the siloxanes[1]. The appearance of the stretching vibration

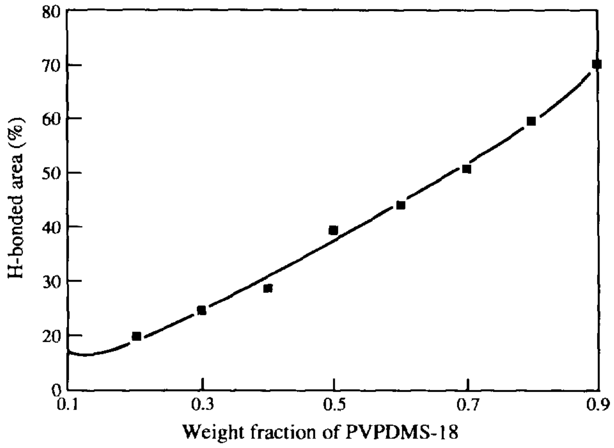

Figure 7 Plots of the fraction of hydrogen bonded amide carbonyl groups as a function of PVPDMS-18 content in the blends

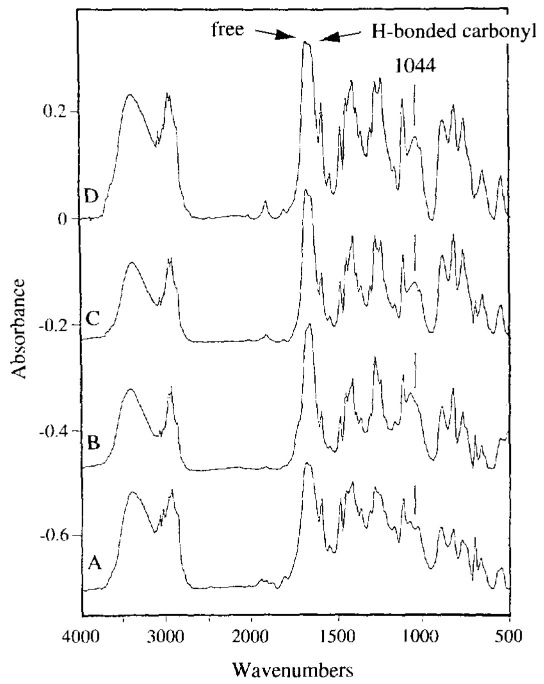

Figure 8 *FT*i.r. spectra of the interpolymer complexes: (A) PVPDMS-34/PVPr; (B) PVPDMS-60/PVPr; (C) PVPDMS-81/PVPr; (D) PVPDMS-100 PVPr

Table 3 Composition and characteristics of the ST-VPDMS/PVPr complexes

		PVPr in feed		Complexes	
No.	Sample	wt%	Si–OH/C=O	Yield (wt%)	Si–OH/C=O^a
1	PVPDMS-34/PVPr	50	0.27	53	0.68
2	PVPDMS-60/PVPr	50	0.41	78	0.66
3	PVPDMS-81/PVPr	50	0.50	82	0.66
4	PVPDMS-100/PVPr	50	0.57	83	0.54

^{a} Determined from elemental analysis

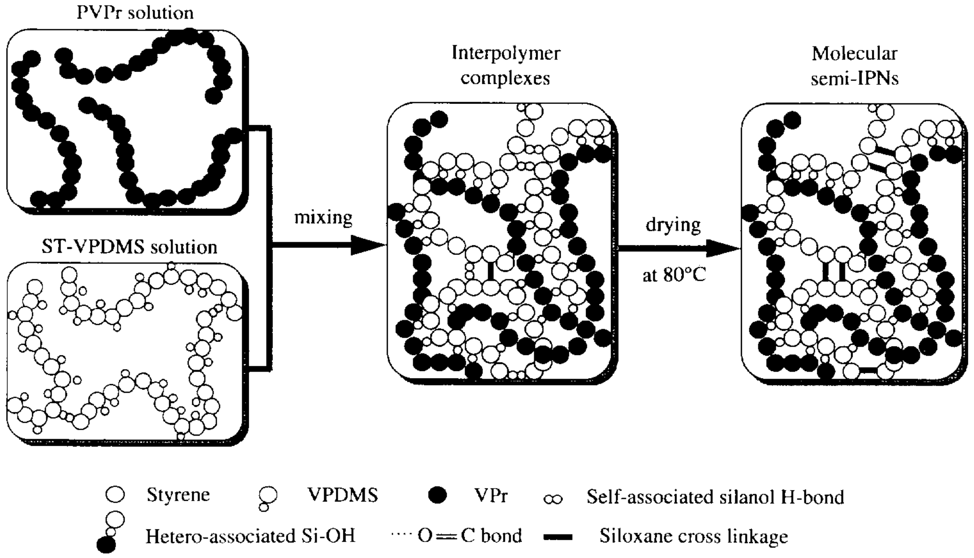

Figure 9 Schematic representation of the formation of molecular-level semi-IPNs from the ST–VPDMS/PVPr complexes

band of Si–O–Si at 1044 cm^{-1} in the infra-red spectra was an indication of formation of the siloxanes by condensation of the silanols. In the infra-red spectra of the interpolymer complexes (*Figure 8*), we have noted that the Si–O–Si stretching vibration band appeared for the PVPDMS-60/PVPr, PVPDMS-81/PVPr and PVPDMS-100/PVPr complexes, but was barely detectable for the PVPDMS-34/PVPr complex. The appearance of the Si–O–Si stretching vibration band suggested that some of the self-associated dimethylsilanol groups in the complexes were converted into siloxane crosslinkages to form semi-interpenetrating polymer networks (semi-IPNs) by condensation of the dimethylsilanol groups when the VPDMS content exceeded 60 mol% in the copolymers.

A schematic representation of the formation of ST–VPDMS/PVPr semi-IPNs is presented in *Figure 9*. From the infra-red spectroscopic study we knew that the strength of the self-associated hydrogen band was comparable to that of the intermolecular hydrogen bond, and thereby there was still a large fraction of self-associated dimethylsilanol groups in the complexes. A small number of the self-associated silanol groups were probably already converted into siloxane crosslinkages during the isolation of the precipitates as a result of the spontaneous condensation of the dimethylsilanol groups, thus resulting in the formation of molecular-level penetrated polymer networks (*Figure 9*). In the subsequent drying process, more self-associated dimethylsilanol groups were transformed into siloxane crosslinkages.

The temperature (80°C) at which the complexes were dried was chosen to ensure the removal of the solvent on the one hand, and to be below the glass transition temperature of the blends on the other hand, so that the intermolecular hydrogen bond between the silanol and the amide carbonyl groups remained almost intact[17]. The higher glass transition temperature of PVPDMS-60 could be attributed to the propensity of intermolecular hydrogen bonds and the formation of siloxane crosslinkages in the complex. The disappearance of glass transition temperatures in the case of the PVPDMS-81 and PVPDMS-100 complexes might suggest that the semi-IPNs obtained have higher crosslinking densities, consisting of contributions from both the siloxane chemical crosslinkages and the physical crosslinkages of the strong intermolecular hydrogen bond between the silanol and the amide carbonyl groups, which results in very small changes in the heat capacity (ΔC_p) between the glass and rubbery states.

Interpenetrating polymer networks (IPNs) are an important category of polymer materials, with properties that can be quite different from their component networks. When only one of the component polymers is crosslinked, a semi-IPN is obtained. There has been ample documentation in the literature on this subject[18–20]. However, most of the IPNs and related materials that have been investigated to date are phase-separated, and only a limited number of examples of miscible IPNs have been reported[21–24]. In the study of the formation of interpenetrating polymer networks from miscible hydrogen bonded polymer blends, Kim *et al.*[17] demonstrated that the formation of miscible or phase-separated semi-IPNs depended on whether the temperature at which the crosslinking reaction was conducted lay either above or below the lower critical solution temperature (*LCST*). In the silanol-containing polymer blends, the silanol functional group not only acts as a strong hydrogen donor, thus enhancing polymer–polymer miscibility, but also as a chemical crosslinker to form siloxane crosslinkages in the solid state by the condensation of the self-associated silanols. The silanol content in the copolymers and the relative strengths of the hetero- and self-associated hydrogen bonds in the blends are the two key characteristics for the successful preparation of these molecular-level semi-IPNs.

CONCLUSIONS

The study of poly(styrene-*co*-4-vinylphenyldimethylsilanol) and poly(*N*-vinylpyrrolidone) blends showed that approximately 4 mol% of the VPDMS functional group in the styrene copolymer could achieve miscibility with PVPr. Infra-red spectroscopy studies indicated that strong intermolecular hydrogen bonding interaction occurred between the amide carbonyl groups of the PVPr and the dimethylsilanol groups of the ST–VPDMS copolymers. This strong intermolecular hydrogen bond resulted in the formation of interpolymer complexes in the form of mutual precipitates with copolymers containing more than 34 mol% VPDMS. Semi-interpenetrating polymer networks (semi-IPNs) were prepared from the interpolymer complexes by condensation of the self-associated dimethylsilanol groups for copolymers containing more than 60 mol% VPDMS.

The present studies have not only demonstrated that the silanol group was very effective in promoting polymer–polymer miscibility but also explored a new approach for the preparation of semi-IPNs in selected miscible hydrogen bonded polymer blends by complexation of the two component polymers and subsequent condensation of the self-associated silanol groups without the presence of any external crosslinkers or catalysts.

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