The Phase Behavior and Thermal Stability of Blends of Poly(styrene-*co*-methacrylic acid)/Poly(styrene-*co*-4-vinylpyridine)

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ABSTRACT: The hydrogen bonding and miscibility behaviors of poly(styrene-*co*-methacrylic acid) (PSMA20) containing 20% of methacrylic acid with copolymers of poly(styrene-*co*-4-vinylpyridine) (PS4VP) containing 5, 15, 30, 40, and 50%, respectively, of 4-vinylpyridine were investigated by differential scanning calorimetry, thermogravimetric analysis (TGA), and Fourier transform infrared spectroscopy (FTIR). It was shown that all the blends have a single glass transition over the entire composition range. The obtained T_gs of PSMA20/PS4VP blends containing an excess amount of PS4VP, above 15% of 4VP in the copolymer, were found to be significantly higher than those observed for each individual component of the mixture, indicating that these blends are able to form interpolymer complexes. The FTIR study reveals presence of intermolecular hydrogen-bonding interaction between vinylpyridine nitrogen atom and the hydroxyl of MMA group and intensifies when the amount of 4VP is increased in PS4VP copolymers. A new band characterizing these interactions at 1724 cm^{-1} was observed. In addition, the quantitative FTIR study carried out for PSMA20/PS4VP blends was also performed for the methacrylic acid and 4-vinylpyridine functional groups. The TGA study confirmed that the thermal stability of these blends was clearly improved. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 462–468, 2011

Key words: differential scanning calorimetry (DSC); thermogravimetric analysis (TGA); Fourier transform infrared spectroscopy (FTIR); interpolymer complexes; polymer blend

INTRODUCTION

During the last 30 years, research of new polymer materials with improved properties has been the focus of intense interest from both academic and industrial points of view. It is well known that the miscibility of polymer blends is mainly dependent on the value of mixing enthalpy because the contribution of mixing entropy is negligible. In general, to obtain a miscible blend system, it is usually necessary to guarantee that favorable specific interaction exists between the components of the blends.[1–6]

However, two polymers, which initially form an immiscible system, could be enhancing their compatibility by introducing the complementary chemical structures capable to establish specific interactions between their chains. A common strategy is to introduce the specific interactions within two dissimilar constituents by copolymerization. In this context, several types of specific interactions have been reported. Among them, hydrogen bonding plays a vital role in enhancing the physical and thermodynamical properties of polymer materials.[7–9] In this sense, it is reported that the complexation behavior between two polymers containing interacting species strongly depending on the solvent, strength, and the concentration of these species. Meftahi et al. suggested that when methanol used as solvent, the mixture of poly(4-vinylpyridine) with poly(4-hydroxystyrene) formed the insoluble complex. However, they have not observed any formation of interpolymer complexes when pyridine used as solvent.[10] Katime and coworkers have also observed similar behavior with mixture of poly(vinylpyridine) and poly(mono-*n*-alkyl itaconates).[11]

Many techniques can be used to investigate the phase behavior and miscibility of polymer blends. For this purpose and owing to their simplicity and speed, thermal analyses and spectroscopy methods are regularly utilized. These techniques offer information about the miscibility in the different range scales. Hence, the observation of single glass transition for polymer blends determined by DSC makes

evident the homogeneity of the blend on the scale of 20–30 nm. It is reported that the NMR spectroscopy can prove this homogeneity at a molecular scale.[12] Fourier transform infrared spectroscopy (FTIR) has proven to be powerful for the characterization of specific interactions and phase behavior of polymer blends through the observed variation of spectroscopic vibration bands, known as frequency shift.[13,14]

These types of specific interactions have been widely described by the association model theory developed by Painter and coworkers.[8] They suggested the introduction of an additional term into a simple Flory-Huggins expression to account for the free energy of the hydrogen bonding formation.

It is well known that polystyrene (PS) is immiscible with the poly(4-vinylpyridine) (P4VP) and poly(styrene-co-4-vinylpyridine) (PS4VP) in the whole composition range. This behavior has been widely analyzed in detail by some authors. Clarke et al. have concluded that the Flory-Huggins interaction parameter (χ) for blends of PS/P4VP is very large (vary from 1.4 to 7.5).[15] More recently, Ten Brinke and coworkers have demonstrated using mean-field theory that χ is close to 0.3–0.35, reflecting that the presence of strong repulsion interactions occurred between unlike species.[16]

The aim of this article is to explore the miscibility in binary blends by means of DSC, FTIR, and TGA. First, we examined in detail by DSC technique the phase behavior of blends of poly(styrene-co-methacrylic acid)/Poly(styrene-co-4-vinylpyridine). containing 20 mol % of methacrylic acid with several poly(styrene-co-4-vinylpyridine) copolymers, which molar composition vary from 5 to 50% in mol of 4VP. Second, the underlying interactions were studied qualitatively and quantitatively by FTIR. In addition, the relationship between the observed phase behavior of blends as determined by DSC, the formation of hydrogen bonding explored by FTIR, and thermal stability measured by TGA were also investigated.

EXPERIMENTAL

Materials

Poly(styrene-co-methacrylic acid) containing 20 mol % of methacrylic acid and poly(styrene-co-4-vinylpyridine) containing 5, 15, 30, 40, or 50 mol % of 4-vinylpyridine (PS4VP05, PS4VP15, PS4VP30, PS4VP40, and PS4VP50) were synthesized by solution free radical polymerization at 60°C using azo-bis-isbutyronitrile (AIBN) as initiator and chloroform as a solvent. Once the polymerization was complete, the different copolymers were purified by reprecipitation from chloroform solution into an excess of heptane and dried under vacuum pressure for several days.

TABLE I
Characteristics of Copolymers Used in This Work

Polymers	M_w	M_w/M_n	T_g (°C)
PS4VP05	103,000	1.80	105
PS4VP15	134,000	1.60	109.5
PS4VP30	98,500	1.54	114
PS4VP40	143,000	1.77	116.5
PS4VP50	130,000	1.38	122
P4VP	60,000	–	145.3
PSMA20	150,000	1.84	135.8

The methacrylic acid and 4-vinylpyridine contents in the PSMA and PS4VP copolymers were determined by elemental analysis and ^{1}H and ^{13}C NMR. The molecular weights of these copolymers were determined by GPC relative to PS standard. The polydispersities were found in the range of 1.6–1.8. The thermogravimetric measurements were carried out under nitrogen from 50 to 600°C on a TGA-Q500 with a heating rate of 10°C/min. The glass transition temperatures of pure polymers and of their binary blends were measured with a Perkin Elmer DSC PYRIS 1 equipped with an intracooler. All T_g measurements were carried out at a heating rate of 20°C/min under nitrogen flow, and the T_g value was taken as the midpoint of the transition of the second scan. The characteristics of these copolymers are given in Table I.

FTIR measurement was recorded on a Nikolet Avatar 560 FTIR spectrometer, and 64 scans were collected with a spectral resolution of 2 cm^{-1}. Infrared spectra of the polymer blends were obtained by using the conventional NaCl disk method. The films were prepared by casting in THF onto a NaCl disk. All the samples were dried to constant weight in a vacuum oven at 60°C for several days to slowly evaporate the solvent.

RESULTS AND DISCUSSION

DSC study

The miscibility of the polymer blend can be studied by many techniques. Among them, thermal analysis is mainly used in this area. In general, the DSC is the most convenient method to elucidate the miscibility of polymer blends. It has been established that the observation of a single glass temperature transition (T_g) between those of the pure constituents is evidence of their miscibility, whereas the detection of two T_gs corresponding to the pure constituents, respectively, indicating their immiscibility.[17,18]

Systems that are initially immiscible can be transformed to miscible without the presence of any specific interaction when intramolecular repulsions between the comonomers are sufficient. In this way, we examine the effects of the introduction of styrene
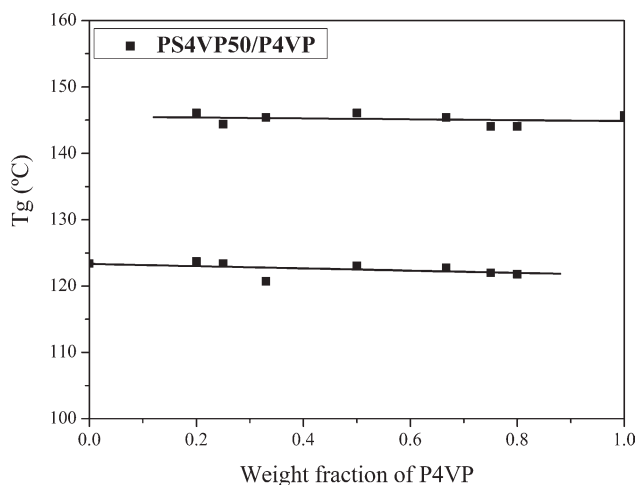
Figure 1 T_g-composition variation versus P4VP weight fraction of PS4VP50/P4VP blends.

comonomer into homopolymer poly(4-vinylpyridine) of an immiscible blends namely PS/P4VP.

As displayed in Figure 1, the inclusion of 50 mol % of vinylpyridine into the homopolymer PS is not sufficient to obtain the miscibility between PS and P4VP. The T_g-composition on PS4VP50/P4VP blends reveals two T_gs for each composition. As can be seen, these glass transition temperatures are similar to those of the pure constituents which are 122 and 145°C, respectively. This result corroborates their immiscibility in the whole composition range. Thus, in this case, the enhancing of the repulsive interactions between comonomers styrene and 4-vinylpyridine is not sufficient to provide the miscibility between the blends of PS4VP50 and P4VP.

The miscibility of PSMA20 with PS4VP05, PS4VP15, PS4VP30, PS4VP40 and PS4VP50, respectively, was investigated by DSC. In these blends, we fixed carboxylic group percentage into PS at 20 mol %, whereas the composition of 4-vinylpyridine into PS4VP was increased gradually. As can be seen in Figure 2, in the case of PSMA20/PS4VP05, a single T_g intermediate between those of the pure constituents PSMA20 and PS4VP05 was observed for each composition. Therefore, the results suggest that the introduction of the carboxylic groups into PS chains enhance the miscibility of PSMA/PS4VP.

Hence, when the composition of 4-vinylpyridine (4VP) in the copolymers PS4VP increases above 15%, the T_g-composition trace has also a positive deviation from the additivity rule. Moreover, the obtained T_gs are higher than of those obtained of the pure constituents in the whole composition range, as occurs in PS4VP40 and PS4VP50 cases. This phenomenon may be attributed to the presence of strong interpolymer interactions. Furthermore, the obtained T_gs suggests that the maximum of the specific interactions moves from ~ 3/1 in

PSMA20/PS4VP15 to 2/1 for PSMA20/PS4VP40 and PSMA20/PS4VP50, respectively.

To quantify the intensity of specific interactions within various PSMA20/PS4VP systems, we applied the Kwei equation developed essentially to describe the T_g of the blends where strong intermolecular interactions are involved, particularly when there is a strong deviation from the additivity rule[17]:

$$T_{g,\text{blend}} = \frac{w_1 T_{g1} + k w_2 T_{g2}}{w_1 + k w_2} + q w_1 w_2 \qquad (1)$$

where $T_{g,\text{blend}}$ is the glass transition temperature of the blend; w_1, w_2, T_{g1}, and T_{g2} are the weight fractions and glass transition temperatures, respectively, of the corresponding constituents. The k and q are adjustable parameters related to the degree of curvature of the T_g-composition plots. The q constant measures the extent of the specific interactions that occurred between the two copolymers.

Figure 3 shows the constant q as a function of a percentage amount of 4VP in corresponding blends. As can be observed, the specific interaction density (q) rises with the increase of the percentage of 4VP in the blends. These results clearly imply that basic sites serve as a physical cross-link in blends and increase their T_gs. Additionally, the introduction of the basic sites may act as precursor to remove the intramolecular hydrogen bonding of the pure methacrylic acid (PSMA20). This evolution confirms the presence of the stronger hydrogen bonding interaction with increasing the amount of 4-vinylpyridine in both blends between unlike species.

FTIR study

FTIR spectroscopy is widely used to explore the presence of specific interactions in the polymer
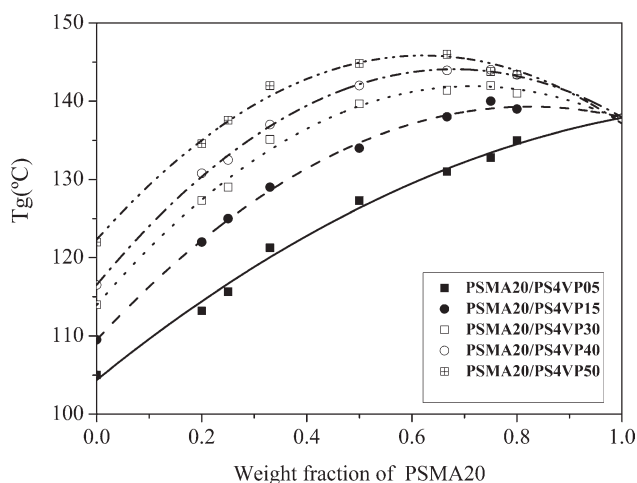
**Figure 2** T_g-composition variation versus PSMA20 weight fraction of PSMA20/PS4VP systems.
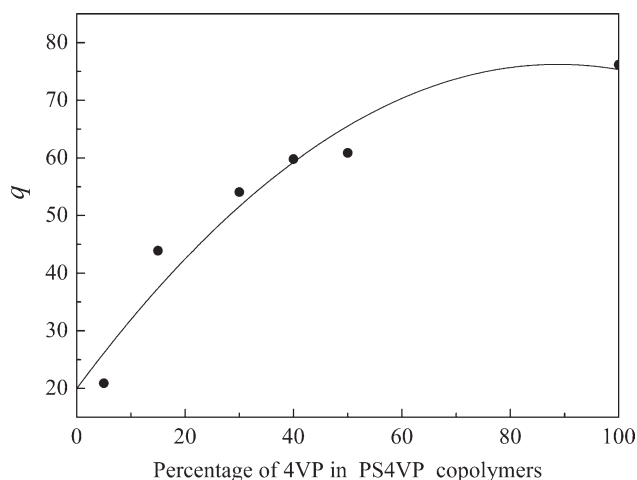
Figure 3 q-composition variation versus percentage of 4VP of PSMA20/PS4VP systems.

blend system. It is well known that hydrogen bonding interactions considerably affect the energy of the covalent bonds on interactive species, thus a frequency shift can be detected. The IR carbonyl-hydroxyl stretching range or flexion mode of pyridine-hydroxyl interactions is mainly sensitive to the hydrogen bonding formation.

The 1560–1620 cm^{-1} region is characterized by one broad band between 1601 cm^{-1} and 1597 cm^{-1} as shown in Figure 4, attributed to styrene and pyridine rings, respectively. The latter band shifts slightly to higher wavenumbers and becomes more intense with increasing PS4VP content in the blend. Similar phenomena are observed in the rest of the PSMA20/PS4VP systems. It is important to note that we have not observed the formation of any pyridinium ion for
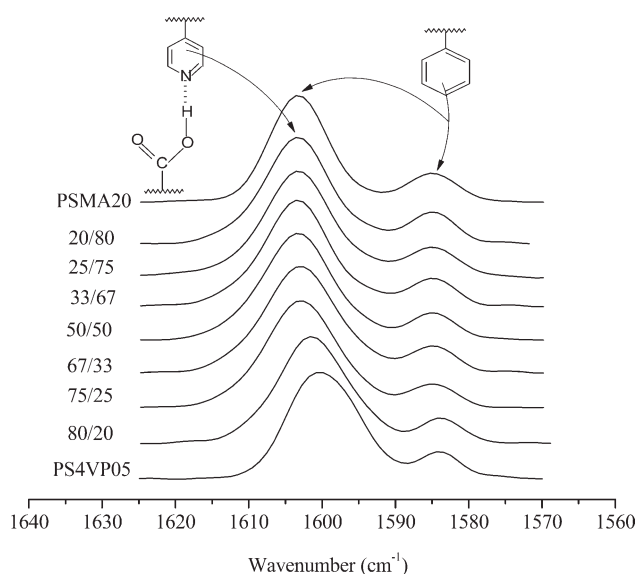
Figure 4 FTIR spectra of PSMA20, PS4VP05, and their blends in 1570–1640 cm^{-1} region.
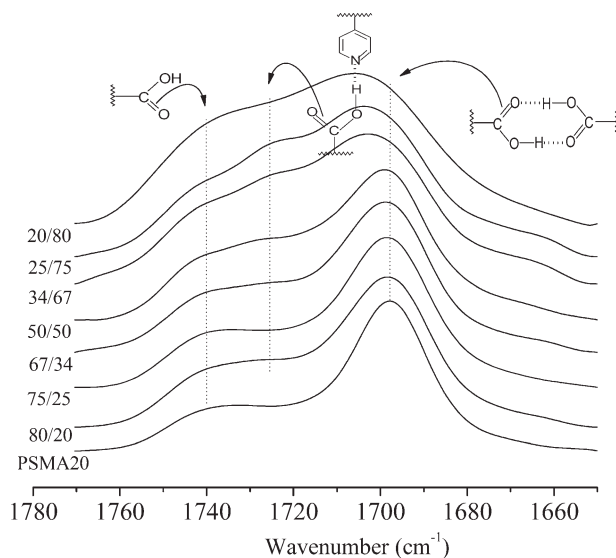

Figure 5 FTIR spectra of PSMA20, PS4VP05, and their blends in 1650–1780 cm^{-1} region.

all systems. This is in good agreement with that suggested previously by Goh and coworkers in complexes of poly (methacrylic acid) and poly(4-vinyl-pyridine).[19] The shift is due to an increase in the rigidity of the pyridine ring and may be also considered as an evidence of the presence of the hydrogen bonding between PSMA20 and PS4VP05.

On the other hand, the carbonyl stretching (1650–1780 cm^{-1}) region is particularly important for two reasons. First, the PS4VP copolymers did not give any band in this region, whereas the pure acidic copolymer (PSMA20) shows two characteristic bands. A free (non-hydrogen-bonded) carbonyl band localized at 1740 cm^{-1} and the self-associated carboxylic acid band localized at 1700 cm^{-1}, the latter band is more extensive than the former. However, the carbonyl stretching may certainly be affected by the presence of intermolecular interactions. For example, Figure 5 shows a scale expanded FTIR spectra in the carbonyl region of PSMA20, PS4VP05, and their blends in different ratios. When PS4VP05 is added, the carbonyl stretching band observed at 1700 cm^{-1} of pure PSMA20 shifted slightly to a higher frequency (up to 1709 cm^{-1}) due to the breaking of the self-association between carboxylic groups. The appearance of a new band is clearly identified at 1724 cm^{-1}, which is assigned to the free carbonyl groups liberated as a consequence of hydrogen bonding formed between hydroxyl and pyridine nitrogen atom.

These results were in good agreement with those obtained previously by Coleman and coworkers.[20] Similar observations have been also found by Akiyama and coworkers in their study of blends of poly(styrene-co-methacrylic acid) including MA units from 4.8 to 27 mol % with poly(ethylene glycol).[21]

I: free carbonyl II: Self association H-bonding III: MA-4VPy H-bonding
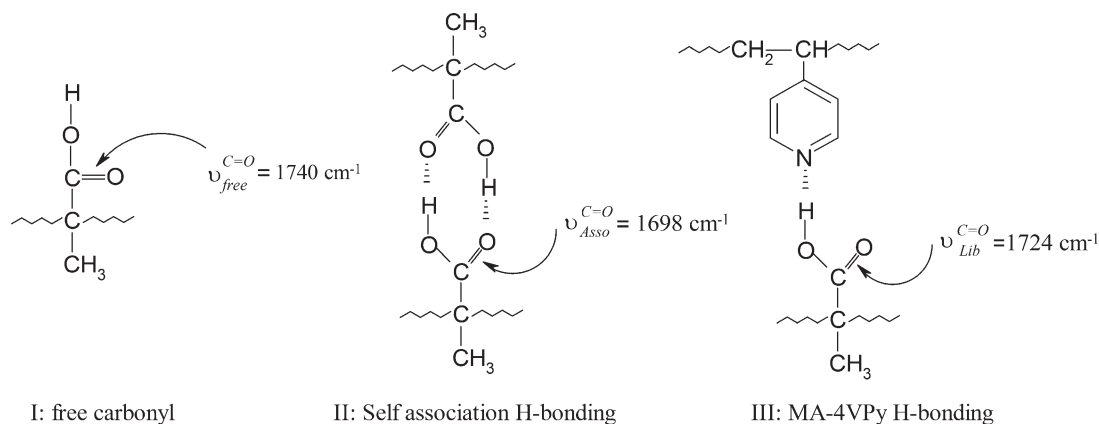
Scheme 1 Potential interactions involved on PSMA20/PS4VP systems in the carbonyl stretching region.

According to the chemical structures of both copolymers, the present polymer blends form associated structures in solution, and the structures could be kept even in bulk state. Thus, the expected interactions within the PSMA20/PS4VP systems are illustrated in Scheme 1, where three different interaction types in carbonyl stretching region involved in these systems can be observed.

The pyridine stretching mode observed at 1597 cm^{-1} is mainly perturbed when the hydrogen bonded interaction is developed. The new band appears at higher frequency, approximately at 1607 cm^{-1}, which is attributed to associated pyridine. Unfortunately, the band at 1597 cm^{-1} is rather difficult to analyze due to its overlap with the 1601 cm^{-1} band of the phenyl groups. Although a quantitative analysis is relatively complex in this region, we determined the fraction of free pyridine using adequate curve fitting by the following equation[13]:

$$f_{\text{free}}^{\text{Pyr}} = \frac{A_{\text{free}}^{\text{Pyr}}}{A_{\text{free}}^{\text{Pyr}} + \left(\frac{a_{\text{Ass}}}{a_{\text{free}}}\right) A_{\text{Ass}}^{\text{Pyr}}} \quad (2)$$

where $A_{\text{free}}^{\text{Pyr}}$ and $A_{\text{Ass}}^{\text{Pyr}}$ are the peak areas corresponding to absorptions from free and associated pyri-

dine, respectively. The absorptivity coefficient (a) is the specific absorption ratio of these bands ($a = a_{\text{Ass}}/a_{\text{free}}$). In our case, we assume that they have the same absorption coefficient, i.e., a equals to unity.

The fractions of free and associated pyridine of PSMA20/PS4VP40 are obtained using an appropriate curve fitting method and are summarized in Table II. The associated pyridine ring fraction increased with increasing the amount of PSMA20 in the blend, whereas the free pyridine fraction decreased. This is an indication of the presence of specific interactions. For example, Figure 6 shows the evolution of free and associated pyridine with the PSMA20 composition for the PSMA20/PS4VP05 and PSMA20/PS4VP30 blends. As can clearly be seen, the associated pyridine fraction is very important in the PSMA20/PS4VP05 blend and decreases progressively with increasing 4VP in PS4VP copolymers. It indicates the ability of basic sites to interact with the carboxylic groups. We believe that these results may be due to the preferential intermolecular interactions between unlike species comparatively to self-associated ones. The dilution of MA and 4VP either segments with inert styrene moieties should increase the intermolecular interactions. The blends with the other PS4VP copolymers showed similar behavior.

TABLE II
Curve Fitting Data from Infrared Spectra of PSMA20/PS4VP40 Blends in the 1570–1640 cm^{-1} Region

PSAM20/PS4VP40	ν (cm^{-1})	$W_{1/2}$ (cm^{-1})	f_{free}(Pyr)	ν (cm^{-1})	$W_{1/2}$ (cm^{-1})	f_{Ass}(Pyr)
80/20	1596.9	10.68	0.575	1607.2	8.00	0.425
75/25	1596.7	10.44	0.627	1607.0	7.62	0.373
67/33	1596.4	10.11	0.676	1606.9	7.40	0.324
50/50	1596.4	10.40	0.727	1607.0	7.21	0.273
33/67	1596.0	9.78	0.791	1605.9	6.90	0.209
25/75	1596.1	9.69	0.843	1606.4	6.73	0.157
20/80	1595.9	9.76	0.878	1605.5	7.00	0.122

$W_{1/2}$, Width at the half-height.
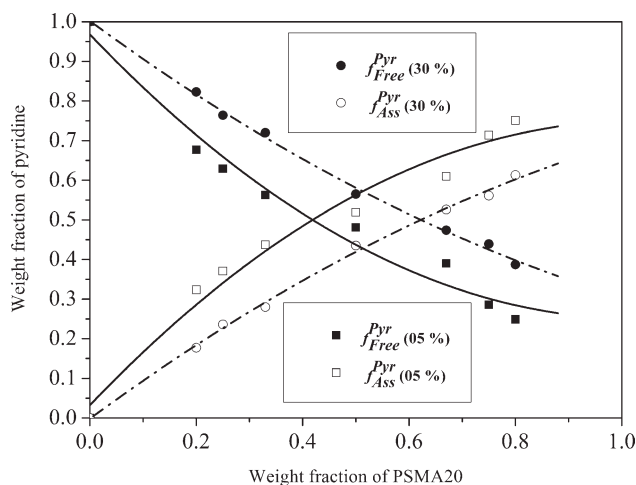
Figure 6 Evolution of free and associated pyridine fractions versus PSMA20 composition of PSMA20/PS4VP05 and PSMA20/PS4VP30 systems.
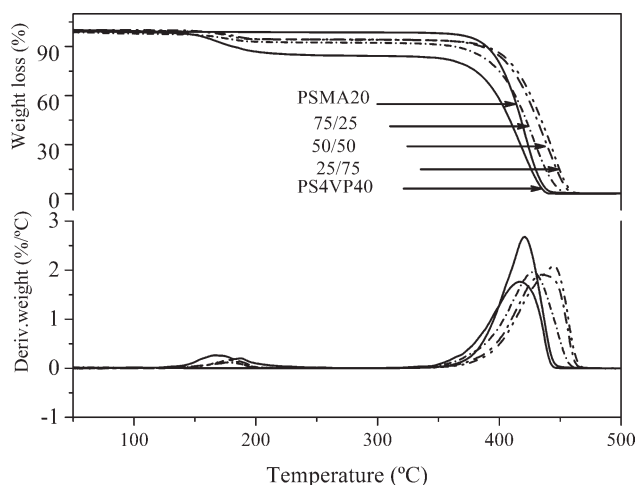

Figure 7 TG and DTG curves for PAM20, PS4VP40, and their blends.

The free, liberated, and associated carbonyl fractions were calculated by the following equation using an adequate curve fitting:

$$f_{\text{free}}^{\text{C=O}} = \frac{A_{\text{free}}^{\text{C=O}}}{A_{\text{free}}^{\text{C=O}} + A_{\text{Lib}}^{\text{C=O}} + aA_{\text{Ass}}^{\text{C=O}}} \quad (3)$$

where A is the peak areas corresponding to free, associated, and liberated carbonyl groups. The ratio absorptivity (a) is assumed equal to 1.1.[13] Table III summarized the three carbonyl fractions for PSMA20/PS4VP40. These results indicate that the liberated carbonyl fraction increases with increasing of the PS4VP40 content in the blend, whereas that of free carbonyls remains fairly constant. Moreover, we believe that the hydrogen bonded interactions occurred between the hydroxyl group of MA and the 4VP nitrogen atom is caused essentially by removing the self associations of the pure PSMA20.

TGA study

The thermal stability of polymer materials constitutes is one of the key factors in their application for several sectors. To determine the upper temperature limit of the processing and use of blends, the thermogravimetric analysis (TGA) is widely used to understand the thermal degradation of polymer blends, miscibility, and the possibility to form crosslinked structures. In this context, the thermal behavior of PSMA20/PS4VP40 was studied.

As reported by several authors that the thermal behavior of poly(methacrylic acid) exhibits two stage degradation process.[22–25] The first stage is assigned to the loss of water molecules through the formation of intra- and intermolecular anhydride links and also to the decarboxylation of a fraction of the carboxylic groups. The second stage is mainly attributed to the decomposition of the main chain by random scissions. The thermogravimetric curves for PSMA20, P4VP40, and their different ratios are shown in Figure 7. It can be observed that PSMA20 shows a good thermal stability until about 350°C. Above this temperature, only one exothermic event corresponding to the decomposition of the polymer was detected. This result could be due to the lower amount of carboxylic groups in PSMA20 and their statistic distribution. Both facts hinder the acidic

TABLE III
Curve Fitting Data from Infrared Spectra of PSMA20/PS4VP40 Blends in the 1650–1780 cm^{-1} Region

PSMA20/PS4VP40	ν (cm^{-1})	$W_{1/2}$ (cm^{-1})	f_{free}	ν (cm^{-1})	$W_{1/2}$ (cm^{-1})	f_{Asso}	ν (cm^{-1})	$W_{1/2}$ (cm^{-1})	f_{lib}
82/20	1740	17	0.081	1699	28	0.615	1724	29	0.303
75/25	1741	15	0.061	1699	28	0.600	1723	32	0.336
67/33	1741	15	0.064	1700	30	0.572	1723	33	0.334
50/50	1741	18	0.032	1700	32	0.545	1723	34	0.423
33/67	1740	14	0.013	1700	32	0.457	1720	34	0.530
25/75	1741	11	0.023	1700	29	0.402	1720	35	0.568
20/80	1740	12	0.023	1700	32	0.402	1720	33	0.575

Samples	Stage I		Stage II	
PSMA20/PS4VP40	T_1 (°C)	% Weight loss	T_2	% Weight loss
PSMA20	98	1.3	420	60
75/25	181	3	444	65
50/50	178	5	435	70
25/75	176	8	427	75
PS4VP40	167	10	416	68

group contacts on the main chain and consequently reducing the formation of the anhydride links.

However, P4VP copolymers showed two well-defined decomposition stages, suggesting a different behavior in terms of thermal stability. In the first stage, a relatively small mass loss was observed in the temperature range 110–240°C, which is assigned to the release of water absorbed by the hydrophilic groups of P4VP. In our previous study, we observed that the percentage mass loss of the PS4VP copolymers increased slightly with increasing 4VP in the copolymer.[26] When the PSMA20 amount was gradually increased in the blend, the relative stability of these blends rose base on the fact that the mass loss observed with pure P4VP in the first stage decreases considerably. It is interesting to point out that all PSMA20/P4VP40 blends showed better improvement thermal stability with the resulting blends. It indicates that the preferential acid–basic interactions were stronger than those occurred between nitrogen atom of pyridine and absorbed water molecules that act as physical croos-links. Table IV summarizes the thermogravimetric parameters of the studied copolymers and their blends in different ratios.

CONCLUSIONS

In this work, the phase behavior of PSMA20 with a variety of PS4VP copolymers has been investigated by DSC, FTIR, and TGA. We showed that depending on the amount of vinylpyridine introduced within PS, miscible blends or complexes are formed due to hydrogen bonding that occurred between PSMA20 and PS4VP. This miscibility or complexation was first evidenced by DSC where a single T_g was detected for each composition of the blends. It has also been shown that the blends of PSMA20 with PS4VP40, PS4VP50, and P4VP had, for each composition, a single T_g higher than those obtained for the pure constituents.

FTIR results also confirm the presence of these hydrogen bonding interactions qualitatively by the appearance of a new band at 1724 cm^{-1}, attributed to liberated carbonyl group by the hydrogen bonding formed between basic and hydroxyl groups, and quantitatively from the evaluation of the fractions of free and associated pyridine and the liberated and the associated carbonyl groups in their corresponding regions.

As a result of strong intermolecular interactions that occurred between unlike species, TGA study for some PSMA20/PS4VP blends show their improvement thermal stability. The changes of the decomposition process are rather than those of the neat copolymers are good indication for their miscibility.

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