

Interpolymer Complexation and Blend Formation between Poly(*N*-vinyl-2-pyrrolidone) and Aliphatic Hydroxyl-Containing Polymers

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(Received September 26, 1994)

ABSTRACT: The interpolymer complexation and blend formation between poly(*N*-vinyl-2-pyrrolidone) (PVP), a tertiary amide polymer, and three aliphatic hydroxyl-containing polymers, namely, poly(2-hydroxypropyl methacrylate), poly(hydroxyether of bisphenol-A) and poly(styrene-*co*-allyl alcohol), has been studied. PVP forms interpolymer complexes with all the three aliphatic hydroxyl-containing polymers in tetrahydrofuran solutions over the entire feed composition range. The glass transition temperatures of the complexes are higher than those calculated from the additivity rule. PVP has a stronger ability in forming interpolymer complexes with aliphatic hydroxyl-containing polymers than the other three tertiary amide polymers, namely, poly(*N*-methyl-*N*-vinylacetamide), poly(*N,N*-dimethylacrylamide), and poly(2-ethyl-2-oxazoline). Infrared spectroscopic studies show that the hydrogen bonding interactions between the carbonyl groups in PVP and the hydroxyl groups are stronger than the self-associations of the hydroxyl-containing polymers. Semiempirical Austin Model 1 (AM1) calculations reveal that the atomic charges of the carbonyl oxygen atoms of all these tertiary amide polymers are about the same, suggesting that steric factor is important in determining their complexation behavior.

KEY WORDS Interpolymer Complexes / Poly(*N*-vinyl-2-pyrrolidone) / Poly(2-hydroxypropyl methacrylate) / Poly(hydroxyether of bisphenol-A) / Poly(styrene-*co*-allyl alcohol) /

We have recently studied the complexation behavior¹⁻⁴ of three isomeric tertiary amide polymers, namely, poly(*N*-methyl-*N*-vinylacetamide) (PMVAc), poly(*N,N*-dimethylacrylamide) (PDMA), and poly(2-ethyl-2-oxazoline) (PEOx), with aliphatic hydroxyl-containing polymers such as poly(2-hydroxypropyl methacrylate) (PHPMA), poly(hydroxyether of bisphenol-A) (phenoxy), and poly(styrene-*co*-allyl alcohol) (SAA). The results show that the nature of solvents, feed composition, and chemical structures of component polymers are important factors affecting complex formation. Fourier transform infrared spectroscopic studies reveal that the driving force

in achieving complexation is the intermolecular hydrogen bonding associations between the hydroxyl groups and the amide carbonyl groups.

We have also reported that poly(*N*-vinyl-2-pyrrolidone) (PVP), another tertiary amide polymer, is miscible with several hydroxyl-containing polymers when cast from *N,N*-dimethylformamide (DMF) solutions. Such hydroxyl-containing polymers include poly(2-hydroxyethyl methacrylate) (PHEMA),⁵ PHPMA,⁵ SAA,⁵ and copolymers of 2-hydroxyethyl methacrylate.^{6,7} PVP forms miscible blends with poly(vinyl alcohol) (PVA)⁸⁻¹⁰ when cast from aqueous solutions, and with

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phenoxy,¹¹ partially hydrolyzed poly(vinyl acetate)¹² and poly(*p*-vinylphenol) (PVPh)¹³ when cast from non-aqueous solvents. However, PVP can form interpolymer complexes with polymers containing strong proton-donating groups such as poly(acrylic acid) (PAA),¹⁴ poly(methacrylic acid) (PMAA),¹⁴ poly(monoethyl itaconate),¹⁵ and phenolic polymers.¹⁶ Since complexation usually does not occur in DMF, it will be of interest to explore if PVP can also form interpolymer complexes with aliphatic hydroxyl-containing polymers in other solvents in which interpolymer complexes are usually formed. In this paper, the complexation behavior of PVP with PHPMA, phenoxy, or SAA in tetrahydrofuran (THF) solutions is reported and compared with the other tertiary amide polymers.

EXPERIMENTAL

Materials

PVP with a reported weight-average molecular weight of 40.0 kg mol⁻¹ was obtained from Scientific Polymer Products, Inc.; its glass transition temperature (T_g) is 160°C. SAA containing 6.5 wt% hydroxyl group (T_g = 48°C) and phenoxy (T_g = 95°C) were also obtained from Scientific Polymer Products, Inc.; their number- and weight-average molecular weights are 1.75 and 3.26, and 20.3 and 30.8 kg mol⁻¹, respectively. PHPMA (T_g = 95°C) was purchased from Polysciences Inc.; its number- and weight-average molecular weights are 24.0 and 32.7 kg mol⁻¹, respectively.

Preparation of Interpolymer Complexes

Interpolymer complexes of PVP with PHPMA, phenoxy or SAA were obtained by mixing their THF solutions at a concentration of 10 g l⁻¹. Precipitates, which were formed upon mixing the solutions, were separated by centrifugation and washed with the solvent. The complexes were then dried *in vacuo* at 90°C for at least two weeks; they were stored in a

desiccator to prevent absorption of moisture. The nitrogen contents of the interpolymer complexes were determined by elemental analysis, using a Perkin-Elmer 2400 elemental analyzer.

Glass Transition Temperature (T_g) Measurements

A Perkin-Elmer DSC-4 differential scanning calorimeter was used to measure the glass transition temperatures of all samples. Since PVP is hygroscopic, each sample was preheated to 180°C and held at that temperature for 10 min to remove any absorbed moisture. The scanning rate was 20°C min⁻¹ and the T_g was taken as the initial onset of the change of slope in the differential scanning calorimetry (DSC) curve. All the T_g values reported were averages of at least three runs.

Fourier Transform Infrared (FTIR) Characterization

Infrared spectra were recorded on a Perkin-Elmer 1725X FTIR spectrophotometer; 64 scans were signal-averaged at a resolution of 2 cm⁻¹. To ensure the absence of moisture and to eliminate thermal history of sample preparation, spectra were recorded at 160°C using a SPECAC high-temperature cell, equipped with an automatic temperature controller, which was mounted in the spectrophotometer. The sample chamber was purged with N₂. Samples for FTIR analyses were prepared by casting the DMF solutions onto KBr discs and then drying *in vacuo* at 90°C for at least two days. The films were sufficiently thin to be within the absorbance range where the Beer-Lambert law is obeyed.

AM1 Calculations

The Austin Model 1 (AM1)¹⁷ is a semiempirical quantum mechanics method which can be used to study the geometrical and electronic structures, conformational energies and charge densities of polymers as well as small molecules.^{18,19} In this work, AM1 calculations

of low molecular weight model compounds of the tertiary amide polymers were carried out using a MOPAC package version 6.0 installed on an IRIS computer. The initial geometric parameters such as bond lengths and bond angles were assigned using the literature data²⁰⁻²² of similar compounds. The initial dihedral angles were assumed based on the internal coordinates. The calculated results of atomic electron charge distributions together with geometric structures were used to interpret the complexation behavior of these tertiary amide polymers.

RESULTS AND DISCUSSION

Complexation between PVP and Aliphatic Hydroxyl-Containing Polymers

PVP forms interpolymer complexes with all the three aliphatic hydroxyl-containing polymers in THF solutions. Data on the yields and compositions of the interpolymer complexes and their T_g 's are listed in Table I.

For the PVP/PHPMA system, complexation occurred at the entire feed composition range. The yields of the complexes were in the range of 46–99 wt%. The compositions of the complexes varied from 45 to 82 mol% of PVP with increasing PVP content in the feed. As shown in Table I, the T_g 's of these complexes vary from 135 to 163°C and are higher than those calculated from the additivity rule, indicating a strong favorable intermolecular interaction between the two polymers.

Similar to the PVP/PHPMA system, PVP formed interpolymer complexes with phenoxy at the entire feed composition range. The yields of the complexes varied from 34 to 92 wt% and were lower than those of the corresponding PVP/PHPMA complexes. This observation can be taken to indicate that the intermolecular interaction between PVP and phenoxy is weaker than that between PVP and PHPMA. The compositions of the complexes, which are highly rich in PVP, are in a relatively narrow range from 73–90 mol% of PVP as compared

Table I. Experimental data of interpolymer complexes between PVP and PHPMA, phenoxy or SAA precipitated from THF solutions

Blend systems	Feed composition		Interpolymer complexes		
	PVP	Content	Yield	PVP content	T_g
	wt%	mol%	wt%	mol%	°C
PVP/PHPMA	20	25	46	45	135
	40	46	80	53	141
	50	57	99	55	145
	60	66	98	65	146
	80	84	98	82	163
PVP/phenoxy	20	39	34	79	152
	40	63	74	73	149
	50	72	82	77	152
	60	79	92	77	152
	80	91	84	90	162
PVP/SAA	20	17	26	55	143
	40	35	44	60	152
	50	44	59	66	155
	60	55	79	67	166
	80	76	93	69	158

to the PVP/PHPMA system. The T_g 's of the complexes are higher than those calculated from the additivity rule.

Complexation also occurred between PVP and SAA over the entire composition range upon mixing their THF solutions. The yields of the complexes varied from 26 to 93 wt% with increasing PVP content in the feed and were generally lower than those of the PVP/PHPMA and PVP/phenoxy systems, demonstrating that SAA has the weakest ability among the three aliphatic hydroxyl-containing polymers in forming complexes with PVP. The compositions of the complexes are also rich in PVP and are in a range of 55–69 mol% of PVP, narrower than the corresponding PVP/PHPMA system. Similarly, the T_g values of the complexes are also higher than those calculated from the additivity rule.

It has been reported that PVP forms miscible blends with PHPMA, phenoxy and SAA in DMF solutions.^{5,11} These results are consistent with the general trend that tertiary amide polymers cannot form complexes with aliphatic hydroxyl-containing polymers in DMF solutions due to the strong hydrogen bond breaking ability of DMF.

Table II summarizes the miscibility and complexation behavior of various tertiary amide polymers. The results show that PVP exhibits a stronger ability in forming interpolymer complexes with aliphatic hydroxyl-containing polymers than the other three tertiary amide polymers.

FTIR Characterization

FTIR is a useful technique to study polymer-polymer interactions. The frequency shifts of the hydroxyl bands of hydroxyl-containing polymers are commonly used to measure the strength of hydrogen-bonding interactions. Since PVP is hygroscopic, it is essential to exclude moisture from the polymer samples. Therefore, all the FTIR measurements were carried out at 160°C.

Figure 1 shows the infrared spectra in the

Table II. Miscibility and complexation behavior between tertiary amide polymers and aliphatic hydroxyl-containing polymers in THF solutions^a

	SAA	Phenoxy	PHPMA
PVP	C	C	C
PMVAc	M*/C* (1)	C (3)	C (4)
PDMA	M (2)	C (3)	C (4)
PEOx	M (2)	M (3)	C (4)

^a C = formed complexes at all feed compositions; M = formed miscible blends at all feed compositions; C* = formed complexes when SAA content in the feed is ≥ 20 wt%; M* = formed miscible blends when SAA content in the feed is 10 wt%. Reference number in parentheses.

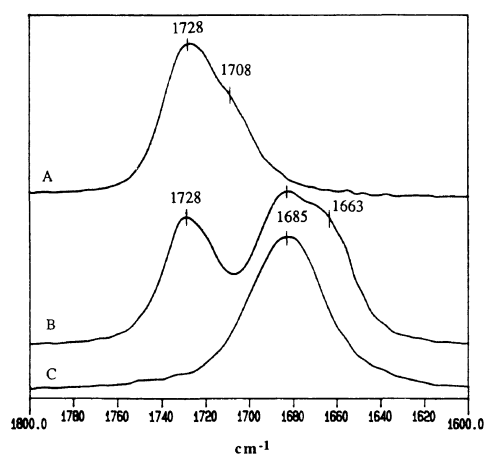


Figure 1. FTIR spectra in the carbonyl region: (A) PHPMA; (B) PVP/PHPMA containing 40 wt% PVP; (C) PVP.

carbonyl stretching region, recorded at 160°C, of DMF-cast PHPMA, PVP, and a PVP/PHPMA blend containing 40 wt% PVP. There are two bands in the carbonyl region of PHPMA (see curve A). The main absorption at 1728 cm^{-1} is attributed to the free carbonyl groups and the shoulder at 1708 cm^{-1} is ascribed to carbonyl groups hydrogen bonded with hydroxyl groups (self-association). PVP (see curve C) has only one absorption at 1685 cm^{-1} attributed to the free carbonyl band of PVP. A new band at 1663 cm^{-1} in the spectrum of the PVP/PHPMA blend (see curve B) can

be reasonably considered as the contribution of the carbonyl groups in PVP which are hydrogen bonded with the hydroxyl groups in PHPMA. Upon increasing the PHPMA content in the blends, the relative intensity of the shoulder at 1663 cm^{-1} increases gradually as shown in Figure 2. In order to further study the extent of hydrogen bonding interactions in PVP/PHPMA blends and their composition dependence, a curve-fitting analysis was conducted for both the ester and amide carbonyl bands, following the methodology of Coleman and co-workers²³ which we employed in our previous studies.⁴

Table III shows the least-squares curve-fitting results of the carbonyl stretching band

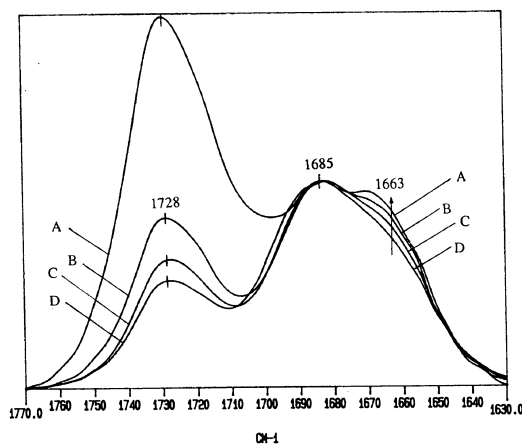


Figure 2. FTIR spectra in the carbonyl region of DMF-cast PVP/PHPMA blends, recorded at 160°C : (A) 20; (B) 40; (C) 50; (D) 60 wt% of PVP.

of the PVP/PHPMA blends. The fraction of hydrogen bonded ester carbonyl groups in PHPMA, f_B (PHPMA), was calculated by the following equation (eq 1) using an absorptivity ratio, a_F/a_B , of 1.23 obtained by Garton²⁴ for the inter-association between the hydroxyl groups in phenoxy and the ester carbonyl groups in poly(ϵ -caprolactone):

$$f_B = (a_F/a_B)A_B / [A_F + (a_F/a_B)A_B] \quad (1)$$

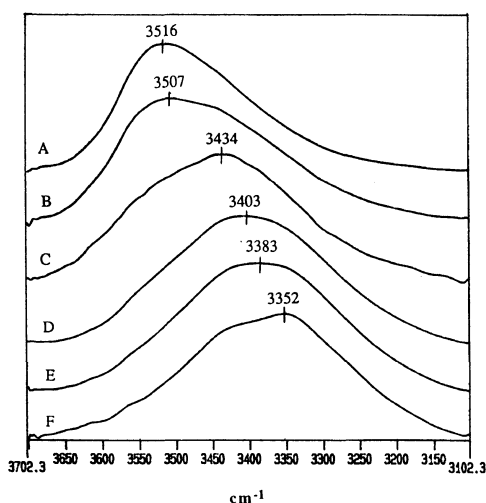
where A_F and A_B are the areas of the free (1728 cm^{-1}) and hydrogen-bonded (1708 cm^{-1}) carbonyl bands, and a_F and a_B are the absorptivities of the two bands. The fraction of hydrogen-bonded amide carbonyl groups (see Table IV) was again calculated from eq 1 using an absorptivity ratio, a_F/a_B , of 1.3 for the amide carbonyl groups in PDMA inter-associated with the hydroxyl groups in PVPh.²⁵ For pure PHPMA at 160°C , 44% of the carbonyl groups are hydrogen-bonded with hydroxyl groups. Upon mixing with PVP, the fraction of hydrogen-bonded ester carbonyl groups in PHPMA decreases drastically with increasing PVP content in the blend up to 50 wt% of PVP, and the fraction becomes too small to be detected when PVP content in the blends is over 60 wt%. This result shows that the amide carbonyl groups in PVP is a stronger proton-acceptor in hydrogen bonding with the hydroxyl group than the ester carbonyl groups in PHPMA. Thus, the quantitative analysis confirms that the intermolecular hydrogen

Table III. Curve-fitting results of the ester carbonyl band in PVP/PHPMA blends

Blend composition		Free C=O band			H-bonded C=O band			Fraction of H-bonded C=O, $f_B/\%$
PVP	Content	Freq	Width	Area,	Freq	Width	Area,	
wt%	mol%	cm^{-1}	cm^{-1}	A_F	cm^{-1}	cm^{-1}	A_B	
0	0	1728	26	22	1708	28	14	44
20	25	1728	31	18	1707	19	2.0	12
40	46	1728	29	14	1707	17	0.90	7.3
50	57	1728	27	9.7	1708	13	0.39	4.7
60	66	1727	28	8.0	1708	—	—	0
80	84	1726	25	23	1708	—	—	0

Table IV. Curve-fitting results of the amide carbonyl band in PVP/PHPMA blends

Blend composition		Free C=O band			H-bonded C=O band			Fraction of H-bonded C=O, $f_B/\%$
PVP wt%	Content mol%	Freq cm^{-1}	Width cm^{-1}	Area, A_F	Freq cm^{-1}	Width cm^{-1}	Area, A_B	
20	25	1686	25	6.8	1663	27	6.5	55
40	46	1686	27	14	1663	26	11	51
50	57	1686	30	16	1662	27	9.0	42
60	66	1687	32	17	1662	28	8.1	38
80	84	1687	35	18	1663	27	6.5	32

**Figure 3.** FTIR spectra in the hydroxyl region of DMF-cast PVP/PHPMA blends, recorded at 160°C: (A) 0; (B) 20; (C) 40; (D) 50; (E) 60; (F) 80 wt% PVP.

bonding interaction between the hydroxyl groups in PHPMA and the amide carbonyl groups in PVP is stronger than the self-association between the hydroxyl and the ester carbonyl groups in PHPMA. On the other hand, the amide carbonyl groups in pure PVP are not hydrogen bonded. Upon the addition of PHPMA, the fraction of hydrogen-bonded amide carbonyl groups in PVP increases steadily (Table IV).

Similarly, the addition of phenoxy or SAA to PVP also leads to the development of a shoulder at 1663 cm^{-1} , and the intensity of the shoulder band increases with increasing phe-

noxy or SAA content.

Figure 3 illustrates the hydroxyl region of DMF-cast PVP/PHPMA blends recorded at 160°C. PHPMA shows a broad band centered at 3516 cm^{-1} , attributed to the overlapped bands of free hydroxyl groups and hydroxyl groups which are hydrogen bonded to other hydroxyl groups and carbonyl groups. With increasing PVP content in the blends, the center of the band shifts gradually to lower frequencies, indicating that the intermolecular hydrogen bonding interaction between PVP and PHPMA is stronger than the self-association of PHPMA.

Figures 4 and 5 show the hydroxyl stretching region of DMF-cast PVP/phenoxy and PVP/SAA blends recorded at 160°C. The free and hydrogen bonded hydroxyl bands of phenoxy and SAA are discernible. With increasing PVP content in the blends, the intensities of free hydroxyl bands of phenoxy at 3570 cm^{-1} and those of SAA at 3590 cm^{-1} decrease, indicating that more free hydroxyl groups are now involved in intermolecular associations with the carbonyl groups. Meanwhile, the centers of the broad hydrogen bonded bands shift gradually to lower frequencies when the PVP content in the two blend systems increases, demonstrating that more hydroxyl groups are involved in intermolecular hydrogen bonding interactions with PVP carbonyl groups. The magnitude of the frequency shift of phenoxy hydroxyl groups hydrogen bonded with

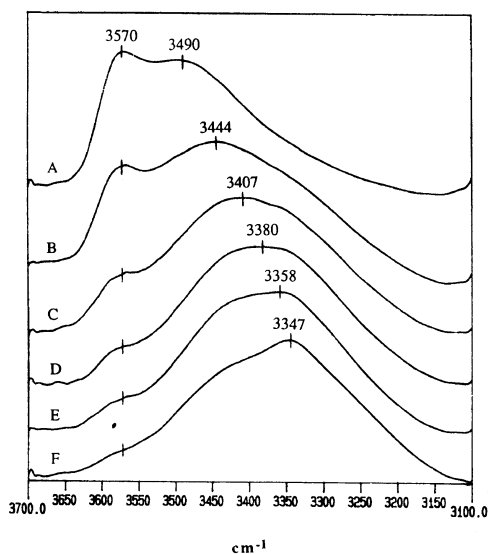


Figure 4. FTIR spectra in the hydroxyl region of DMF-cast PVP/phenoxy blends, recorded at 160°C: (A) 0; (B) 20; (C) 40; (D) 50; (E) 60; (F) 80 wt% PVP.

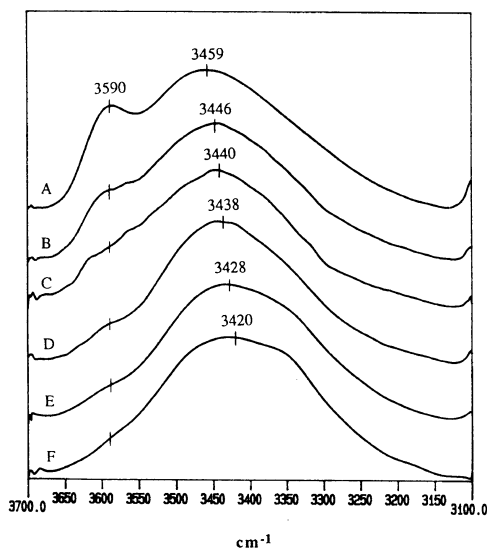


Figure 5. FTIR spectra in the hydroxyl region of DMF-cast PVP/SAA blends, recorded at 160°C: (A) 0; (B) 20; (C) 40; (D) 50; (E) 60; (F) 80 wt% PVP.

carbonyl groups in PVP with respect to the free hydroxyl groups ($\Delta\nu = 223 \text{ cm}^{-1}$), which can be used as a measure of the average strength of the intermolecular interactions,¹³ is larger than that of SAA hydroxyls ($\Delta\nu = 170 \text{ cm}^{-1}$), in-

Table V. Atomic charges of oxygen and nitrogen atoms of solvents and model compounds of tertiary amide polymers

Polymer/Solvent	O	N
PVP	-0.349	-0.337
PMVAc	-0.370	-0.340
PDMA	-0.366	-0.332
PEOx	-0.371	-0.342
THF	-0.284	—
DMF	-0.365	-0.353

dicating a stronger intermolecular hydrogen bond association between PVP and phenoxy than that between PVP and SAA. This trend is consistent with the complexation abilities of the two blend systems as discussed earlier. The frequency shifts of PVP blends are also higher than the corresponding blends of PMVAc, PDMA, or PEOx,¹⁻⁴ showing that PVP does interact more strongly with hydroxyl-containing polymers as compared with the other three tertiary polymers.

AMI Calculations

The present series of studies has established that the complexation abilities of the four tertiary amide polymers are in the order of PVP > PMVAc > PDMA > PEOx. What are the factors governing the complexation ability? Since infrared spectroscopic studies have shown the existence of hydrogen bonding interactions between the amide carbonyl groups and the hydroxyl groups, could the complexation abilities be related to the atomic charge densities of the carbonyl oxygen atoms of the various tertiary amide polymers? To answer the question, we used the semiempirical AM1 program to calculate the atomic charge distributions of these polymers, using their hydrogenated monomers as model compounds. *N,N*-dimethylpropionamide was chosen as the model compound for PEOx. The calculation results are given in Table V.

There are two possible proton-accepting sites in each tertiary amide polymer—the carbonyl

oxygen and the nitrogen atoms. Evidence from model studies favors the former.^{26,27} As shown in Table V, AM1 calculation results show that the atomic charges of oxygen atoms are more negative than those of nitrogen atoms, indicating that oxygen would be the favored proton-accepting site. However, the atomic charges of oxygen atoms of the three model compounds of PMVAc, PDMA, and PEOx are nearly the same and are more negative than that of the model compound of PVP. Apparently, the atomic charge of the carbonyl oxygen atom is not the only factor determining the complexation ability. It is likely that steric effect can be an important factor. The carbonyl groups in PDMA and PEOx are only one bond length away from the main chains while those in PVP and PMVAc are two bond lengths away. Thus, the carbonyl groups in PDMA and PEOx are less accessible to the proton-donating polymers, giving rise to weaker intermolecular interactions and thereby poorer complexation abilities.

The atomic charge distributions of THF and DMF were also calculated and shown in Table V. The atomic charge of oxygen atom of THF is less negative than those of the model compounds of tertiary amide polymers, indicating that THF cannot complex effectively with tertiary amide polymers for hydroxyl groups. The strong interactions between tertiary amide polymers and hydroxyl-containing polymers lead to the formation of interpolymer complexes in THF. On the other hand, the atomic charge of oxygen atom of DMF is comparable to those of the model compounds of tertiary amide polymers. As a result, DMF is able to interact strongly with hydroxyl groups, preventing the formation of interpolymer complexes.

CONCLUSION

1. PVP forms interpolymer complexes with PHPMA, phenoxy and SAA in THF solutions over the entire feed composition range.

2. PVP interacts more strongly with hydroxyl-containing polymers as compared with PMVAc, PDMA, and PEOx as shown by the frequency shifts of the hydroxyl bands of DMF-cast blends.

3. Semiempirical AM1 calculations suggest that the complexation abilities of tertiary amide polymers are not solely determined by the charge densities of the carbonyl oxygen atoms.

Acknowledgments. Financial support of this work by the National University of Singapore is gratefully acknowledged. We also thank Dr. A. Rai-Chaudhuri for her assistance in the AM1 calculations.

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