Complexation Between Poly(2-hydroxypropyl methacrylate) and Three Tertiary Amide Polymers

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SYNOPSIS

The complexation behavior between poly(2-hydroxypropyl methacrylate) (PHPMA) and three isomeric tertiary amide polymers, namely, poly(N-methyl-N-vinylacetamide) (PMVAc), poly(N,N-dimethylacrylamide) (PDMA), and poly(2-ethyl-2-oxazoline) (PEOx), was studied. PHPMA forms interpolymer complexes with the three tertiary amide polymers at the entire feed composition range in methyl ethyl ketone and tetrahydrofuran solutions, indicating a strong intermolecular interaction between different polymer chains. However, complexation does not occur between PHPMA and the three tertiary amide polymers in N,N-dimethylformamide solutions. Infrared spectroscopic studies show that the intermolecular hydrogen bonding association between the hydroxyl groups in PHPMA and the amide carbonyl groups in the tertiary amide polymers is stronger than the self-association between the hydroxyl groups and the ester carbonyl groups in PHPMA. PHPMA has a stronger complexation ability with these tertiary amide polymers than poly(styrene-co-allyl alcohol) and poly(hydroxyether of bisphenol-A) do. © 1994 John Wiley & Sons, Inc.

INTRODUCTION

Polymers containing strong proton-donating groups, such as carboxylic and phenolic hydroxyl groups, can form interpolymer complexes with polymers containing proton-accepting groups through cooperative hydrogen bonding under certain conditions. Typical examples include poly(acrylic acid) (PAA)/poly(N-vinylpyrrolidone) (PVP),[1] poly(methacrylic acid)/PVP,[1,2] PAA/poly(vinyl methyl ether) (PVME),[3] PAA/poly(2-ethyl-2-oxazoline) (PEOx),[4,5] poly(p-vinylphenol) (PVPh)/poly(N,N-dimethylacrylamide) (PDMA),[6] PVPh/PEOx,[6] and phenol-formadehyde resin/PDMA[7] complexes.

Recently, we investigated the complexation behavior between aliphatic hydroxyl-containing polymers, such as poly(styrene-co-allyl alcohol) (SAA)[8,9] and poly(hydroxyether of bisphenol-A) (phenoxy),[10] and three tertiary amide polymers. We found that interpolymer complexation could occur in methyl ethyl ketone (MEK) or tetrahydrofuran (THF) solutions but not in N,N-dimethylformamide (DMF) solutions. The abilities of these tertiary amide poly-

mers to form complexes with SAA and phenoxy were found to be on the order of poly(N-methyl-N-vinylacetamide) (PMVAc) > PDMA > PEOx. In this article, we present our results on the complexation behavior of poly(2-hydroxypropyl methacrylate) (PHPMA) with PMVAc, PDMA, and PEOx.

EXPERIMENTAL

Materials

Poly(2-hydroxypropyl methacrylate) (PHPMA) was purchased from Aldrich Chemical Company, Inc. The commercially available monomeric 2-hydroxypropyl methacrylate usually contains 20–30% of 1-methyl-2-hydroxyethyl methacrylate. PMVAc and PDMA were synthesized in our laboratory as described previously.[8,9] PEOx was provided by Dow Chemical Company. The number- and weight-average molecular weights and glass-transition temperatures of the polymers are compiled in Table I.

Preparation of Polymer Complexes and Blends

Interpolymer complexes between PHPMA and the three isomeric tertiary amide polymers were ob-

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Table I Characteristics of Polymers

Polymer	M_n	M_w	T_g (°C)
Poly(2-hydroxypropyl methacrylate) (PHPMA)	24000	32700	95
Poly(N-methyl-N-vinylacetamide) (PMVAc)	3300	3700	136
Poly(N,N-dimethylacrylamide) (PDMA)	12000	19600	118
Poly(2-ethyl-2-oxazoline) (PEOx)	38500	61500	56

tained through mixing solutions, each containing 0.1 g of polymer/10 mL of MEK or THF. The precipitates were separated by centrifugation, washed with solvent, and then dried *in vacuo* at 90°C to constant weight. For blends prepared by solution casting from DMF solutions, initial removal of solvent was done on a hot plate at about 90–120°C, and the blends were then dried *in vacuo* at 90°C for at least 2 weeks. All complexes and blends were stored in a desiccator to prevent the absorption of moisture. The nitrogen contents of polymer complexes were determined by elemental analysis using a Perkin–Elmer 2400 elemental analyzer.

Glass-Transition Temperature (T_g) Measurements

The T_gs of the complexes and blends were measured by a Perkin–Elmer DSC-4 differential scanning calorimeter. As tertiary amide polymers are highly hygroscopic, all the samples were preheated to 150°C and held at that temperature for 15 min to ensure complete removal of any moisture and residual sol-

vent. The scanning rate was 20°C min^{-1} and T_g was taken as the initial onset of the change of slope in the differential scanning calorimetry (DSC) curve. All the reported T_g values are the averages of several 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}. Spectra recorded at elevated temperatures were obtained by using a SPECAC high-temperature cell equipped with an automatic temperature controller mounted on the spectrophotometer. The sample chamber was purged with N_2. Samples for FTIR analysis were prepared by casting the DMF solutions onto KBr discs and then dried *in vacuo* at 90°C for at least 2 days. The films were sufficiently thin to be within the absorbance range where the Beer–Lambert law is obeyed.

RESULTS AND DISCUSSION

Complexation Behavior

PHPMA formed interpolymer complexes with all three tertiary amide polymers in MEK and THF solutions. Data on the yields and compositions of the complexes, and their T_gs are listed in Tables II–IV.

For the PHPMA/PMVAc system, interpolymer complexes occurred at the entire feed composition range in both MEK and THF solutions. The yields of the complexes were in the range of 62.3–98.6 wt % and 82.2–98.4 wt % from MEK and THF solutions, respectively, and were higher than those of the SAA/PMVAc[8] and phenoxy/PMVAc[10] systems. The results can be taken to indicate that the intermolecular interaction between PMVAc and PHPMA is stronger than those between PMVAc and SAA or phenoxy. For the complexes obtained from MEK solutions, their compositions varied from

**Table II Experimental Data of PHPMA/PMVAc System**

Solvent	Feed Composition PMVAc Content Wt %	Feed Composition PMVAc Content Mol %	Polymer Complexes Yield (wt %)	Polymer Complexes PMVAc Content (mol %)	Polymer Complexes T_g (°C)
MEK	20	26.7	62.3	31.4	118
MEK	40	49.2	95.1	43.8	120
MEK	50	59.3	97.1	59.4	128
MEK	60	68.6	98.6	68.2	133
MEK	80	85.1	79.8	82.6	139
THF	20	26.7	82.2	40.2	125
THF	40	49.2	93.4	45.8	129
THF	50	59.3	98.4	59.3	132
THF	60	68.6	98.2	66.8	137
THF	80	85.1	97.8	84.7	146

Table III   Experimental Data of PHPMA/PDMA System

| Solvent | Feed Composition PDMA Content | | Polymer Complexes | | |
	Wt %	Mol %	Yield (wt %)	PDMA Content (mol %)	T_g (°C)
MEK	20	26.7	46.9	45.0	110
MEK	40	49.2	88.2	50.7	110
MEK	50	59.3	94.2	54.0	113
MEK	60	68.6	93.7	66.8	116
MEK	80	85.1	65.2	82.1	120
THF	20	26.7	38.1	39.5	115
THF	40	49.2	88.3	41.2	115
THF	50	59.3	96.3	53.0	116
THF	60	68.6	89.9	61.2	118
THF	80	85.1	54.1	63.6	118

31.4 to 82.6 mol % of PMVAc. The T_g values of the complexes are in the range of 118–139°C, which are higher than those weight-average values of the T_gs of the pure polymers as shown in Figure 1. For the complexes obtained from THF solutions, their compositions ranged from 40.2 to 84.7 mol % of PMVAc. The T_g values are in the range of 125–146°C.

Similar to the PHPMA/PMVAc system, PHPMA also formed complexes with PDMA at the entire feed composition range in both MEK and THF solutions. As shown in Table 3, the yields of the complexes were in the range of 46.9–94.2 wt % and 38.1–96.3 wt %, and were lower than those of the corresponding PHPMA/PMVAc complexes, especially when the feed was highly rich in one of

Table IV   Experimental Data of PHPMA/PEOx System

| Solvent | Feed Composition PEOx Content | | Polymer Complexes | | |
	Wt %	Mol %	Yield (wt %)	PEOx Content (mol %)	T_g (°C)
MEK	20	26.7	74.2	30.4	83
MEK	40	49.2	95.9	47.7	76
MEK	50	59.3	98.0	56.6	72
MEK	60	68.6	87.7	61.2	70
MEK	80	85.1	50.4	72.2	68
THF	20	26.7	48.5	42.3	80
THF	40	49.2	87.3	46.4	75
THF	50	59.3	89.1	53.7	73
THF	60	68.6	79.1	56.8	71
THF	80	85.1	38.8	60.9	71
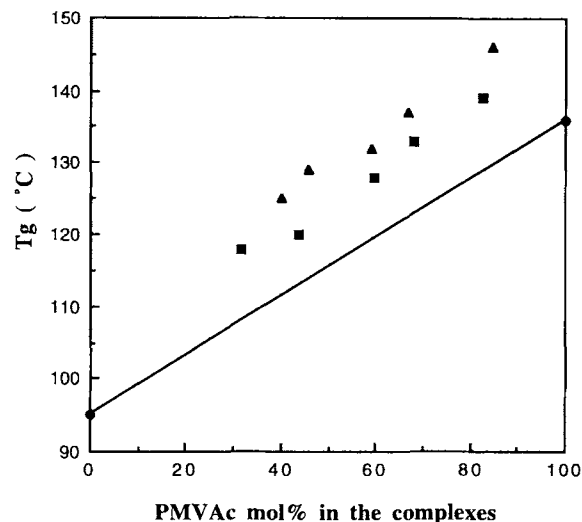
Figure 1 T_g composition relations of the complexes between PHPMA and PMVAc from (■) MEK and (▲) THF solutions.

the component polymers. The results demonstrate that the intermolecular interaction between PHPMA and PDMA is weaker than that between PHPMA and PMVAc. The compositions of the complexes, which depended on feed compositions, varied from 45.0 to 82.1 mol % and 39.5 to 63.6 mol % of PDMA for the complexes obtained from MEK and THF solutions, respectively. The T_g values of the complexes from MEK solutions are in the range of 110–120°C, and those from THF solutions are within 115–118°C. In both cases, the T_g values are higher than those calculated from the additivity rule as shown in Figure 2.
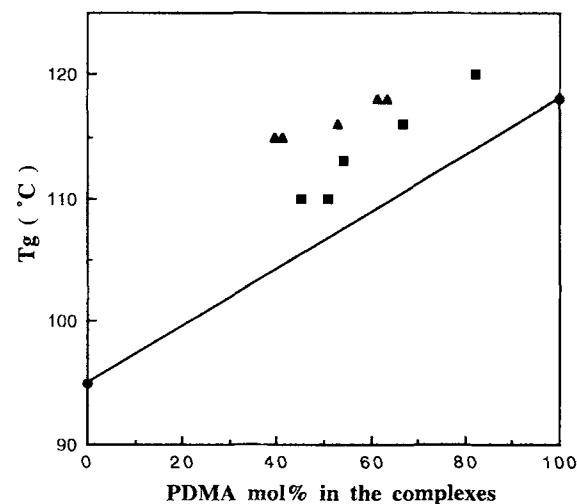
Figure 2 T_g composition relations of the complexes between PHPMA and PDMA from (■) MEK and (▲) THF solutions.

We have earlier reported that both SAA and phenoxy do not form complexes with PEOx in THF, MEK, and DMF solutions. In contrast, PHPMA forms complexes with PEOx at the whole feed composition range in both MEK and THF solutions. The complexation behavior provides additional evidence of the stronger ability of PHPMA in forming interpolymer complexes with tertiary amide polymers as compared with SAA and phenoxy. For the complexes obtained from MEK solutions, the yields of the complexes were in the range of 50.4–98.0 wt %. With increasing PEOx content in the feed, the compositions of complexes changed from 30.4 to 72.2 mol % of PEOx and the corresponding T_g values decreased from 83 to 68°C. For the complexes obtained from THF solutions, the yields of the complexes varied from 38.8 to 89.1 wt %, lower than those obtained from MEK solutions. By increasing the PEOx content in the feed, the PEOx content in the complexes increased from 42.3 to 60.9 mol % and the T_g values of complexes decreased from 80 to 71°C. As shown in Figure 3, the T_g values of the complexes are close to those calculated from the additivity rule.

In common with the general trend, PHPMA did not form interpolymer complexes with the three tertiary amide polymers in DMF solutions because DMF is a strong hydrogen bond breaking solvent. The T_g composition curves of PHPMA/PMVAc (Fig. 4), PHPMA/PDMA (Fig. 5), and PHPMA/PEOx blends (Fig. 6) cast from DMF solutions can be simulated by the Kwei equation[11-12]:
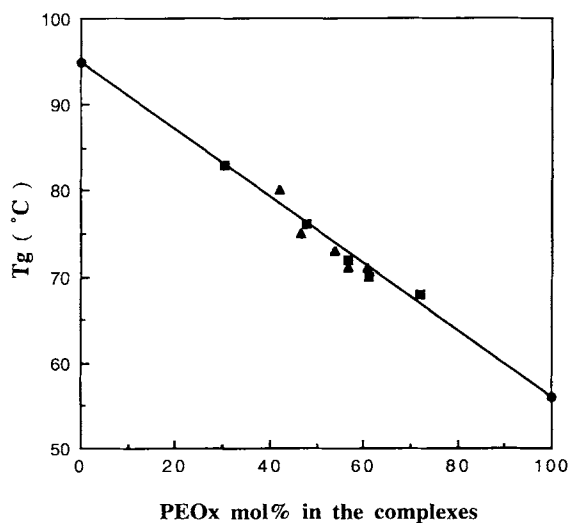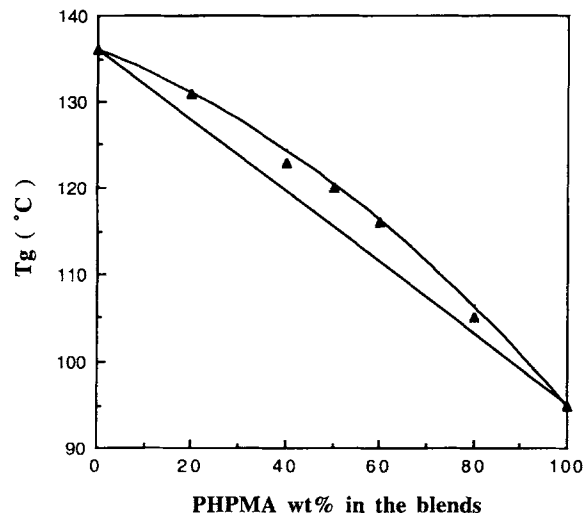

Figure 5 T_g composition curve of PHPMA/PDMA miscible blends cast from DMF solutions.

$$T_g(\text{blend}) = (w_1 T_{g1} + k w_2 T_{g2})/$$
$$(w_1 + k w_2) + q w_1 w_2 \quad (1)$$

where k and q are fitting constants. For the PHPMA/PMVAc blends, the T_g values are higher than those calculated by the additivity rule, and the curve can be fitted with $k = 1$ and $q = 20$. For the PHPMA/PDMA blends, the T_g composition curve is "S-shape" in form and can be fitted with $k = 4$ and $q = 25$. For the PHPMA/PEOx blends, the T_g values are lower than the corresponding calculated weight-average values, and the curve can be fitted with $k = 2$ and $q = 0$. Positive deviations of T_gs for
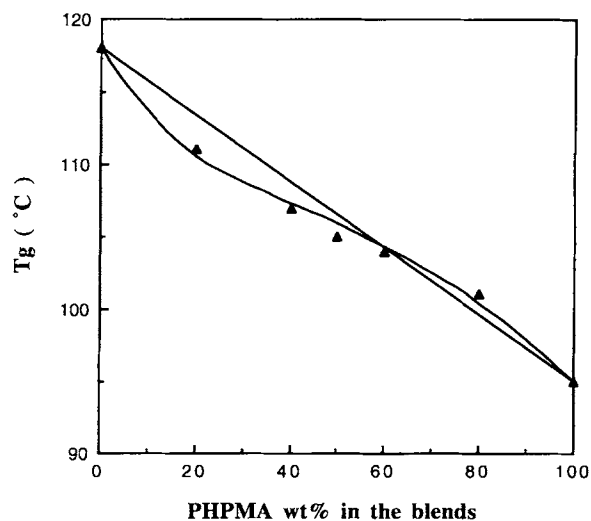

Figure 3 T_g composition relations of the complexes between PHPMA and PEOx from (■) MEK and (▲) THF solutions.

Figure 4 T_g composition curve of PHPMA/PMVAc miscible blends cast from DMF solutions.
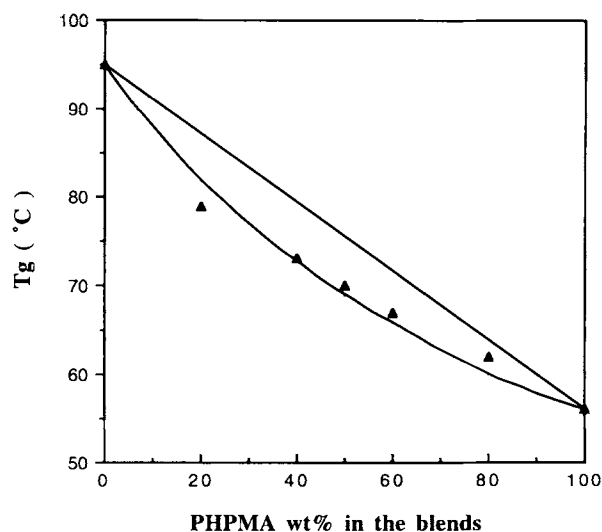
Figure 6 T_g composition curve of PHPMA / PEOx miscible blends cast from DMF solutions.

the blends and the complexes are attributed to the strong intermolecular interactions between the two component polymers that act as physical cross-links, thus reducing chain mobility. In this respect, the three T_g composition curves can then be taken to indicate that the order of the strength of interactions is PMVAc > PDMA > PEOx. It is also noted that the T_g values of the miscible blends are lower than those of the complexes with the same compositions.

FTIR Characterization

Figure 7 shows the infrared spectra in the carbonyl stretching region, recorded at 140°C, of DMF-cast

PHPMA, PMVAc, and a PHPMA/PMVAc blend containing 40 wt % PMVAc. There are two bands in the carbonyl region of PHPMA (see curve A). The "free" carbonyl band is centered at 1728 cm^{-1} and the carbonyls hydrogen-bonded with hydroxyl groups (self-association) are manifested as a shoulder at 1708 cm^{-1}. PMVAc (see curve C) has a strong absorption at 1644 cm^{-1} attributed to the free carbonyl band of PMVAc. A new band at 1629 cm^{-1} is observed in the spectrum of the PHPMA/PMVAc blend containing 40 wt % of PMVAc (see curve B) that is regarded as the amide carbonyl groups in PMVAc hydrogen-bonded with the hydroxyl groups in PHPMA (intermolecular association). As shown in Figure 8, an increase in PMVAc content in the blends leads to a decrease in the relative intensity of the hydrogen-bonded carbonyl band of PHPMA at 1708 cm^{-1} as compared with the free carbonyl peak at 1728 cm^{-1}, indicating that the self-association between the hydroxyls and carbonyls in PHPMA was giving way to the intermolecular association between the hydroxyl groups in PHPMA and the amide carbonyl groups in PMVAc. This suggests that the amide carbonyl groups form stronger hydrogen bonds with hydroxyl groups than the ester carbonyl groups.

Figures 9 and 10 depict the composition effect on the absorbance in the carbonyl region of the PHPMA/PDMA and PHPMA/PEOx blends cast from DMF solutions and recorded at 140°C. The relative intensities of the shoulder at 1708 cm^{-1} contributed from the hydrogen-bonded carbonyl groups in PHPMA decrease upon increasing the content of PDMA or PEOx in the two blend systems.
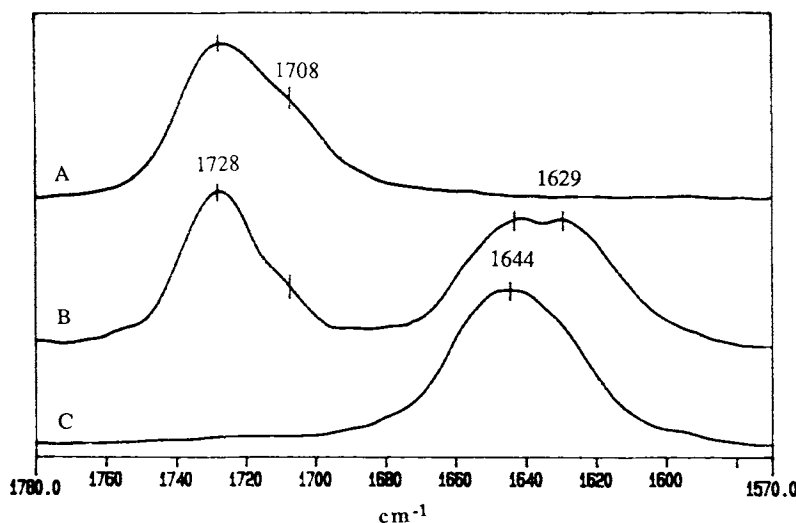

Figure 7   FTIR spectra, recorded at 140°C, of the carbonyl region: (A) PHPMA; (B) PHPMA/PMVAc containing 40 wt % PMVAc; and (C) PMVAc.
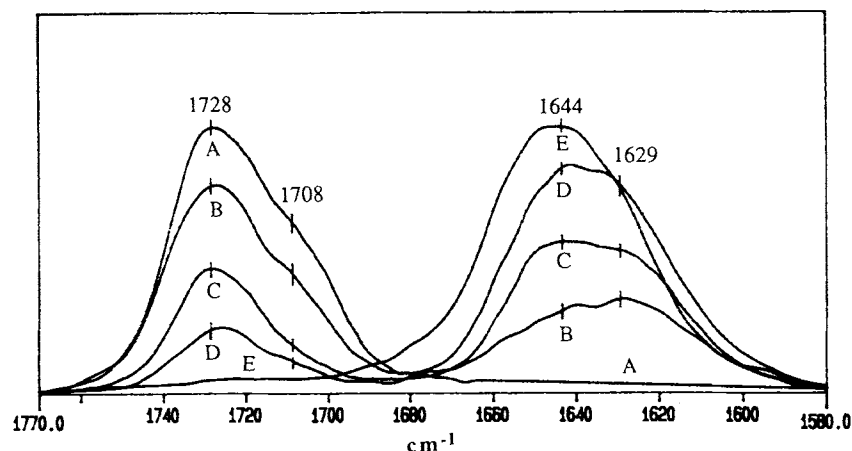
Figure 8   FTIR spectra, recorded at 140°C, of the carbonyl region of PHPMA/PMVAc miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 50; (D) 80; and (E) 100 wt % of PMVAc.

In order to further understand the extent of hydrogen bonding interactions in the PHPMA/PMVAc blends, we conducted a curve-fitting analysis in the carbonyl region of the spectra. As shown in Figure 7, there are four bands in the carbonyl region of PHPMA/PMVAc blends corresponding to the free and the hydrogen-bonded carbonyls of PHPMA and PMVAc. The curve fitting was performed separately for the PHPMA carbonyl band ($1780–1680$ cm^{-1}) and the PMVAc carbonyl band ($1680–1570$ cm^{-1}) following the methodology of Coleman and coworkers.[13,14]

Table V shows the least-squares curve-fitting results of the PHPMA carbonyl stretching band. The fraction of hydrogen-bonded carbonyl groups in PHPMA, f_B, was calculated by the following equation using an absorptivity ratio, a_F/a_B, of 1.23 obtained by Garton[15] for the interassociation between the hydroxyl groups in phenoxy and the ester carbonyl groups in poly(ε-caprolactone):

$$f_B = (a_F/a_B)A_B/[A_F + (a_F/a_B)A_B] \qquad (2)$$

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. For the pure PHPMA at 140°C, 44.8% of the carbonyl groups are hydrogen bonded with hydroxyl groups. Upon mixing with PMVAc, the fraction of hydrogen-bonded ester carbonyl groups in PHPMA drops sharply when the PMVAc content in the blends increases from 0 to 50 mol % and re-
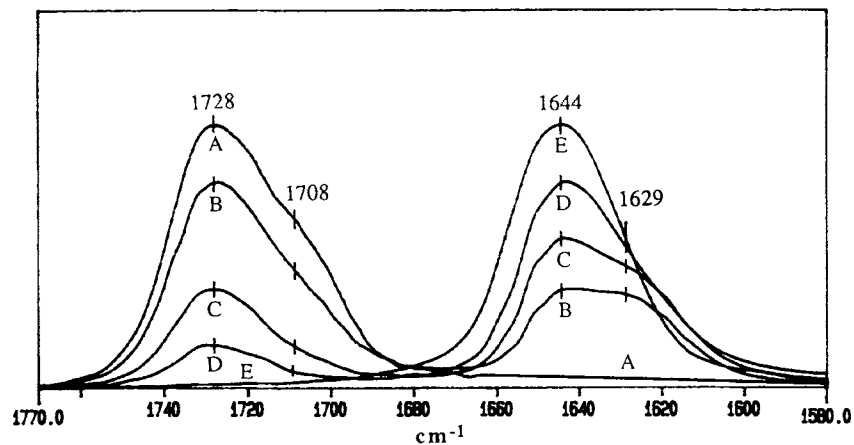

Figure 9   FTIR spectra, recorded at 140°C, of the carbonyl region of PHPMA/PDMA miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 50; (D) 80; and (E) 100 wt % of PDMA.
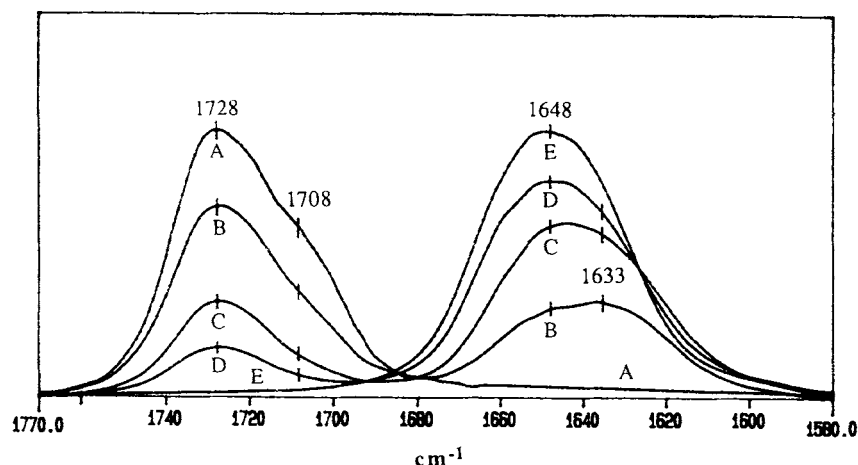
Figure 10 FTIR spectra, recorded at 140°C, of the carbonyl region of PHPMA/PEOx miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 50; (D) 80; and (E) 100 wt % of PEOx.

mains approximately the same when the PMVAc content in the blends is over 50 mol %. The results imply that the amide carbonyl groups in PMVAc are a stronger proton acceptor for hydrogen bonding with hydroxyl groups as compared with the ester carbonyl groups in PHPMA.

Table VI shows the least-squares curve-fitting results of the PMVAc carbonyl stretching band. The fraction of hydrogen-bonded amide carbonyl groups was again calculated from eq. (2) using an absorptivity ratio, a_F/a_B, of 1.3, quoted from Yang et al.,[7] for the amide carbonyl groups in PDMA interassociated with the hydroxyl groups in PVPh. The results show that the extent of intermolecular hydrogen bonding association between the hydroxyl groups and the amide carbonyl groups is quite high especially in those PHPMA-rich blends. It is worthwhile to note that, for the blend containing 40 wt % of PMVAc, the concentration of the ester carbonyl

groups is almost the same as the amide carbonyl groups, but the fraction of the hydrogen-bonded amide carbonyl groups (f_B = 66.1%) is about 2.6 times of the fraction of the hydrogen-bonded ester carbonyl groups (f_B = 25.1%). Thus, this quantitative analysis confirms that the hydrogen bonding interaction between the hydroxyl groups in PHPMA and the amide carbonyl groups in PMVAc (intermolecular association) is stronger than that between the hydroxyl and the ester carbonyl groups in PHPMA (self-association).

The spectra of hydroxyl stretching bands can also be used to characterize the hydrogen bonding interaction between component polymers. Figures 11–13 illustrate the hydroxyl regions of PHPMA/PMVAc, PHPMA/PDMA, and PHPMA/PEOx blends cast from DMF solutions and recorded at 140°C. In contrast to SAA and phenoxy, PHPMA shows only one broad band centered at 3512 cm^{-1} for the combined

Table V Curve-Fitting Results of Ester Carbonyl Band in PHPMA/PMVAc Blends

Blend Composition PMVAc Content		Free C=O Band			H-Bonded C=O Band			Fraction of H-Bonded C=O f_B (%)
Wt %	Mol %	Freq. (cm^{-1})	Width (cm^{-1})	Area (A_F)	Freq. (cm^{-1})	Width (cm^{-1})	Area (A_B)	
0	0	1729	25	21.15	1709	28	13.95	44.8
20	26.7	1728	27	26.26	1706	22	7.17	25.1
40	49.2	1728	26	20.32	1706	18	2.27	12.1
50	59.3	1727	24	22.60	1708	17	2.46	11.8
60	68.6	1727	23	22.36	1708	14	2.21	10.8
80	85.1	1726	25	23.31	1708	14	2.24	10.6

Table VI   Curve-Fitting Results of Amide Carbonyl Band in PHPMA/PMVAc Blends

Blend Composition PMVAc Content		Free C=O Band			H-Bonded C=O Band			Fraction of H-Bonded C=O f_B (%)
Wt %	Mol %	Freq. (cm^{-1})	Width (cm^{-1})	Area (A_F)	Freq. (cm^{-1})	Width (cm^{-1})	Area (A_B)	
20	26.7	1650	24	7.00	1627	37	30.72	85.1
40	49.2	1648	27	15.95	1625	33	23.90	66.1
50	59.3	1648	24	15.85	1626	32	23.34	65.7
60	68.6	1646	28	18.90	1625	32	19.04	56.7
80	85.1	1642	36	28.61	1624	36	10.43	32.2

contributions of the hydroxyl groups hydrogen bonded with other hydroxyl groups and the hydroxyl groups hydrogen bonded with the ester carbonyl groups, which indicates the absence of free hydroxyl groups. With an increasing content of tertiary amide polymer in the blends, the center of the band shifts gradually to lower frequencies (Figs. 11–13). Coleman and colleagues used the frequency difference between free hydroxyl groups and those hydrogen-bonded hydroxyl groups as a measure of the average strength of the intermolecular interactions.[14,16] Because the frequency of the free hydroxyl groups of

PHPMA is not available, we used the hydrogen-bonded hydroxyl frequency of pure PHPMA as a reference value ($\nu = 3512$ cm^{-1}) to compare the relative strength of intermolecular interactions between PHPMA and the three isomeric tertiary amide polymers. The results indicate that the average strength of the hydrogen bond between the PHPMA hydroxyl group and the carbonyl group of each of the three tertiary amide polymers is in the order of PHPMA/PMVAc ($\Delta\nu = 123$ cm^{-1}) > PHPMA/PDMA ($\Delta\nu = 86$ cm^{-1}) > PHPMA/ PEOx ($\Delta\nu = 76$ cm^{-1}). This order is in agreement
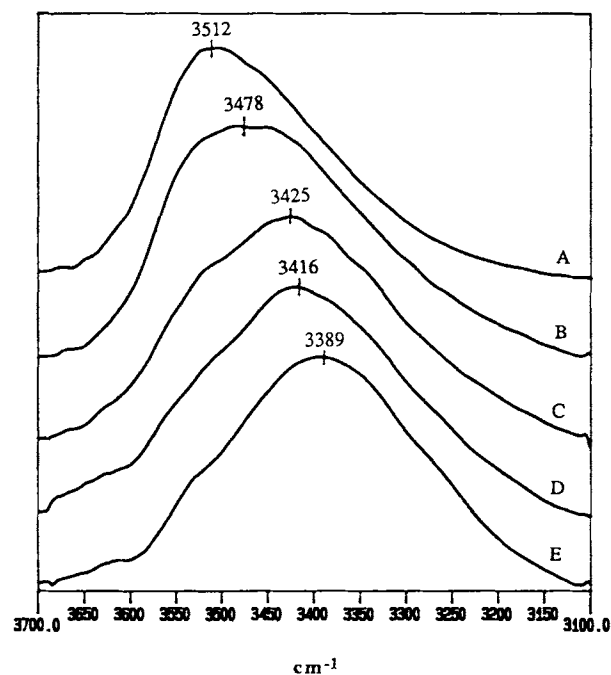

Figure 11   FTIR spectra, recorded at 140°C, of the hydroxyl region of PHPMA/PMVAc miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 40; (D) 50; and (E) 60 wt % PMVAc.
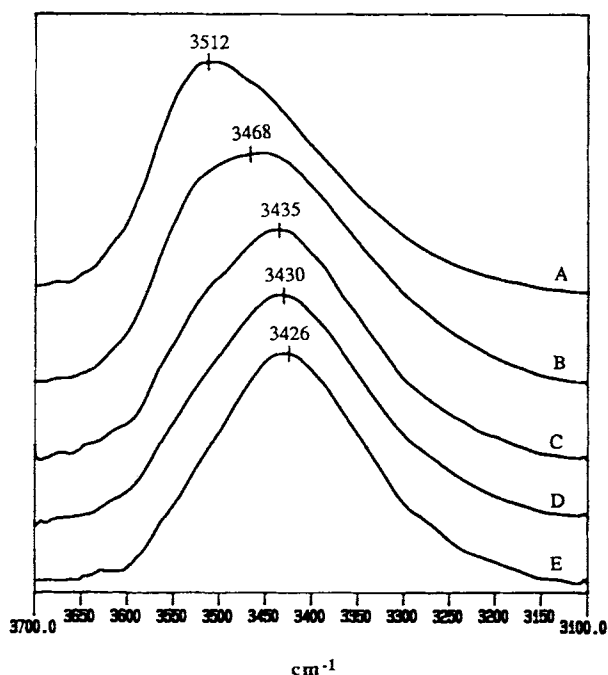
Figure 12   FTIR spectra, recorded at 140°C, of the hydroxyl region of PHPMA/PDMA miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 40; (D) 50; and (E) 60 wt % PDMA.
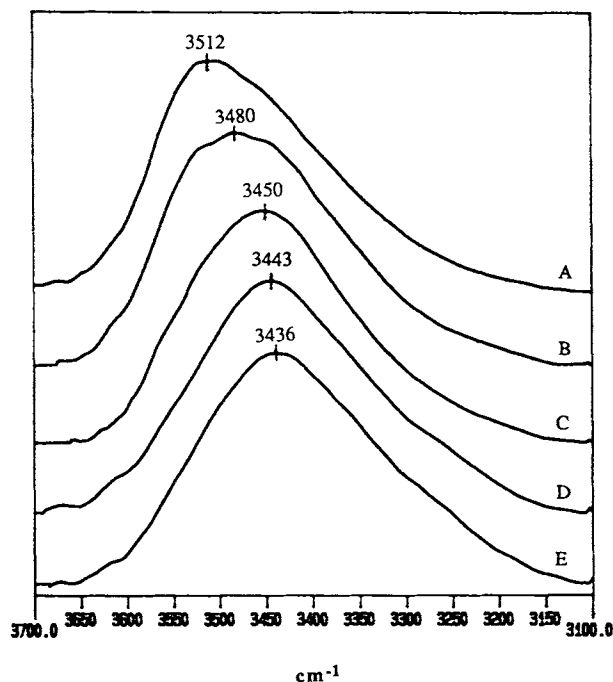
Figure 13 FTIR spectra, recorded at 140°C, of the hydroxyl region of PHPMA/PEOx miscible blends cast from DMF solutions: (A) 0; (B) 20; (C) 40; (D) 50; and (E) 60 wt % PEOx.

with that based on the complexation behavior. The same trend was also observed in our previously reported SAA/tertiary amide polymer[8,9] and phenoxy/tertiary amide polymer[10] systems.

CONCLUSIONS

PHPMA forms interpolymer complexes with PMVAc, PDMA, and PEOX at the entire feed composition range in THF and MEK solutions, but not in DMF solutions. FTIR results indicate that the stronger intermolecular hydrogen bonding association between the hydroxyl groups in PHPMA and the amide carbonyl groups in tertiary amide polymers, as compared with the self-association between the hydroxyl groups and the ester carbonyl groups in PHPMA, is the driving force in forming the in-

terpolymer complexes between PHPMA and the three tertiary amide polymers. PHPMA has a stronger complexation ability with tertiary amide polymers than SAA[8,9] and phenoxy[10] do.

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