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Infrared Spectroscopic Investigation of Chain Conformations and Interactions in P(VDF-TrFE)/PMMA Blends

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ABSTRACT: The blending between poly(methyl methacrylate) (PMMA) and ferroelectric (vinylidene fluoride-trifluorethylene) [P(VDF-TrFE)] copolymer chains has been investigated by Fourier transform infrared (FTIR) spectroscopy over the full range of composition, for the copolymer with 50 mol % of trifluorethylene [TrFE]. The FTIR spectra revealed an absorption band at 1643 cm^{-1}, characteristic of the blend and absent in the individual constituents. We attributed this band to the interaction of the carbonyl group of the PMMA side chains with the disordered helical chains present in the amorphous region of the P(VDF-TrFE). We investigated the consequences of adding PMMA onto the formation of the *all trans* conformation of the copolymer chains and we demonstrated that the effects of thermal heating on the spectra are relevant only for the samples where the ferroelectric semicrystalline phase is present. © 2000 John Wiley & Sons, Inc. J Polym Sci B: Polym Phys 38: 34–40, 2000
Keywords: vinylidene fluoride-trifluorethylene copolymers; polymer blends; ferroelectric phase transition

INTRODUCTION

Since Hill et al.[1] demonstrated the orientation of guest polar molecules by the internal electric field of semicrystalline (vinylidene fluoride-trifluorethylene) copolymers [P(VDF-TrFE)], these materials became good candidates for nonlinear optical (NLO) applications in communication technology.[2] Nevertheless, these ferroelectric copolymers present large light scattering in the UV-visible region because of the presence of small crystallites organized into lamellas, which diffuse the light. Thus, blending with amorphous poly(methyl methacrylate) [PMMA] has been proposed[3,4] as

an efficient method to solve this problem because the good optical properties of PMMA in this spectral range are well known. Binary blends of P(VDF-TrFE)/PMMA were initially investigated in 1986 by Saito et al.,[5] who were interested mainly in their ferroelectric and piezoelectric properties. Later on, Tsutsumi et al.[3] and Bürgel et al.[4] focused on their NLO properties. Recently, we started a systematic investigation of the fundamental properties of these blends in order to contribute to the understanding of their physical behavior. In previous works, we have reported on the kinetics, structural transitions,[6] and dielectric behavior[7] of P(VDF-TrFE)/PMMA blends, demonstrating the full compatibility of the components in the range of 0 to 15 wt % of PMMA, and the partial compatibility above this value until 40 wt % for the copolymer with 50 mol % of

trifluorethylene (TrFE). We also demonstrated that samples with less than 40 wt % PMMA are semicrystalline and that the crystallites present only P(VDF-TrFE) chains in the ferroelectric β form.[6] Although the ferroelectric fraction of the material decreases with increasing PMMA content, the Curie point is not sensitive to the presence of PMMA (the ferroelectric transition occurs always around 65 °C).[6,7]

Thermodynamically, the formation of compatible blends between two polymers results from an attractive interaction between their chains in the melting state. In the case of P(VDF-TrFE)/PMMA blends, we found a relatively high (negative) interaction coefficient, for the particular TrFE composition studied here.[6] Since spectroscopic techniques allow the investigation of particular chemical bonds, we decided to use Fourier transform infrared (FTIR) spectroscopy to study the chain conformations and, possibly, interactions of P(VDF-TrFE)/PMMA blends over a large range of blend composition.

This study is performed through high-temperature spectral analyses in order to take advantage of the information supplied by the ferroelectric phase transition of P(VDF-TrFE),[8–13] which occurs below its melting point. Thus, it could improve our knowledge about the amorphous/semicrystalline blending process, giving additional information to the well-studied PVDF/ PMMA system.[14–20]

EXPERIMENTAL

P(VDF-TrFE) copolymers with 50 mol % of TrFE, supplied by Atochem, were mixed with a commercial type PMMA (Plexiglass™) at compositions of 0, 15, 30, 50, 70, and 100 wt % of PMMA. The blends were prepared dissolving small quantities of the polymer mixture (0,03 g/mL) in a solvent formed by *n,n*-dimethylacetamide (DMAC) and 1 wt % of acetic anhydride at 60 °C. Then, films of about 30 μm were obtained by the conventional casting technique (slow evaporation during one week at 60 °C). A second sample group was prepared by adding a large volume of distilled water to the initial solutions, in order to force fast blend formation. These samples were dried, melted at 200 °C, pressed (300 bar), and quenched to room temperature producing transparent films of about 80 μm.

Transmission infrared spectra were recorded between 40 and 4000 cm^{-1} with a Bomem DA8

spectrometer (Bomem, Inc.), from 25 to 110 °C. The spectral resolution was always better than 2 cm^{-1}. The thicker samples (80 μm) were used below 700 cm^{-1}. Optimized spectra have been obtained by the better choice of accessories: globar source or mercurium arc (above or below 200 cm^{-1}, respectively), MCT or DTGS:PE detector (above or below 800 cm^{-1}), and KBr or Hypersplitter/ Beamsplitter (above or below 400 cm^{-1}). Structural characterization was made by X-ray diffractometry (Rigaku) with 8° 2θ/min scan rate, using CuK$_\alpha$ radiation, in order to confirm the crystallinity degree and the crystalline phase present.

RESULTS AND DISCUSSION

Room-Temperature Analysis

For clarity purposes, we show in Figure 1 the infrared vibrational spectra of P(VDF-TrFE$_{50\%}$)/ PMMA blends only for the 0, 30, and 70 wt % of PMMA samples. In Figure 1(a), we present the results for the samples with about 80 μm, from 400 to 700 cm^{-1} at room temperature. The two most prominent absorption bands at 472 and 506 cm^{-1} correspond to the wagging (w) and bending (δ) vibrational modes of the CF$_2$ dipoles, characteristic of the TT (*trans*) conformation of the ferroelectric β phase of P(VDF-TrFE).[21] A complete mode assignment for the ferroelectric phase of the P(VDF-TrFE) copolymer, based on theoretical and experimental works for the β-PVDF attributions (Cm2m crystallographic group), is given in Table I. It is important to emphasize the one-to-one correspondence between the ferroelectric β structure and the TT chain conformation.

As the PMMA content in the blend increases, the peak intensities of the 472 and 506 cm^{-1} bands first decrease (up to 30 wt %) and then shift (70 wt %) to higher wavenumbers (to 481 and 513 cm^{-1}, respectively). This frequency increase is a first indication of the presence of *gauche* segments in the copolymer chains, since for γ PVDF, where the chains assume a T$_3$G conformation, the w (CF$_2$) and δ (CF$_2$) modes are seen at 481 and 511 cm^{-1}, respectively.[21,22] The absorption peak at 640 cm^{-1} is attributed to the amorphous PMMA.[19] Below 400 cm^{-1}, the spectra do not show any relevant feature (the spectra are not shown here). In fact, they present only two defect-activated copolymer bands: one at 350 cm^{-1}, which can be an IR forbidden twisting mode of CF$_2$ activated by conformational TG defects, and the other at 372 cm^{-1},
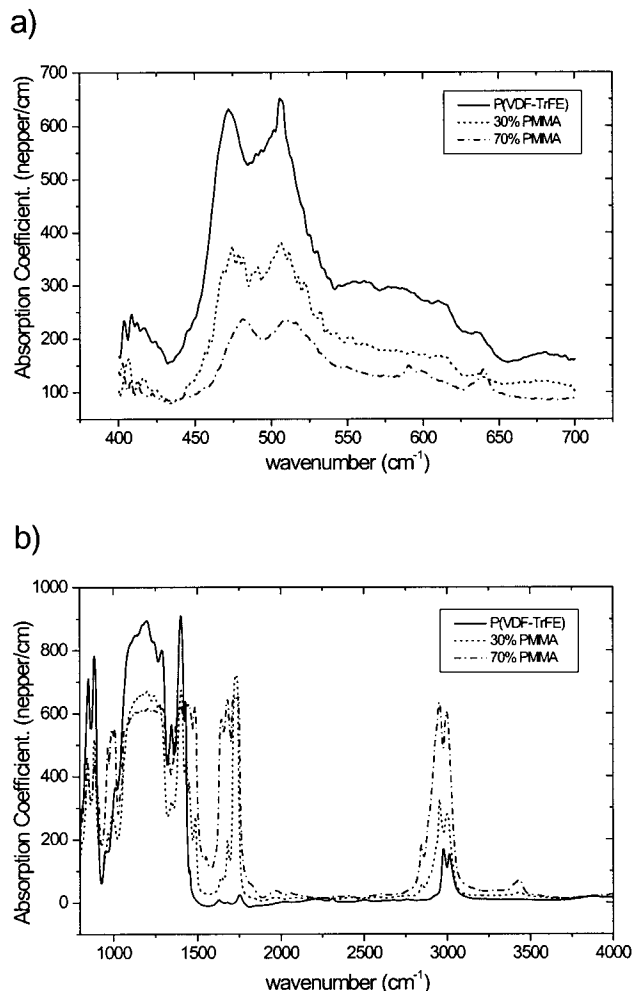
a)

b)

Figure 1. FTIR vibrational spectra for P(VDF-TrFE$_{50\%}$)/PMMA blends in the far (a) and middle infrared (b) region.

which is attributed to head-to-head defects.[22] The librational lattice mode located at 70 cm^{-1} in β PVDF has not been discerned in our spectra. We can attribute its absence to two reasons. First, according to Petzelt et al.,[23] its frequency decreases with increasing TrFE content, and we estimate that for 50 mol % TrFE, it should appear below 50 cm^{-1}. Second, this mode is very sensitive to the crystalline order, which is rather poor in the copolymers with higher TrFE contents.

Figure 1(b) reports the absorption coefficients in the 800 to 4000 cm^{-1} spectral range for the samples with about 30 μm thickness. The most important phenomenon seen here is the peak at 1643 cm^{-1}, which can be observed only for the blends. It is not present in the vibrational spectra of the pure constituents. This can be better ob-

served in Figure 2, which reports a zoom of the 1500–2000 cm^{-1} spectral range and, in the insert, the evolution of the absorption coefficient at 1643 cm^{-1}, with the PMMA content in the blend ($\Delta\alpha$ is the difference between the total absorption coefficient of the 1643 cm^{-1} band and the tail of the 1679 cm^{-1} neighbor band). Note that the absorption is more pronounced for higher PMMA contents.

In order to investigate the influence of the PMMA chains on the formation of the ferroelectric crystalline phase of P(VDF-TrFE), we have plotted the absorption coefficient corresponding to the band at 851 cm^{-1} as a function of the PMMA contents. This band is characteristic of the β PVDF (see Table I) and, as seen in Figure 3, it gradually decreases as PMMA is added to the copolymer, disappearing between 30 and 50 wt % PMMA.

High-Temperature Analysis

P(VDF-TrFE$_{50\%}$) is a ferroelectric copolymer with a first-order ferro-to-paraelectric phase transition around 65 °C, as measured by differential scanning calorimetry (DSC).[6] Thus, because this phenomenon is characteristic of the crystalline regions of the copolymer, we have conducted FTIR investigations in the temperature range of 25–110 °C, with a furnace specially manufactured for this task. This procedure permits us to study the crystallinity and the structure of the blends of P(VDF-TrFE$_{50\%}$)/PMMA by looking at the behavior of the TT and TG conformational chains, before and after the phase transition.

In Figure 4 we show the absorption coefficients for the 851 (a) and 1291 (b) cm^{-1} absorption bands for P(VDF-TrFE$_{50\%}$)/PMMA blends, with 0, 15, and 30 wt % of PMMA (semicrystalline blends) in the 25 to 110 °C temperature range. These two bands are expected to suffer the effects of the ferroelectric phase transition of P(VDF-TrFE), since they are respectively linked to the internal CH$_2$ and CC symmetrical stretching modes that are particularly sensitive to the chain conformation.[21,24,25] As it can be seen in Figures 4(a) and 4(b), there is a clear decrease in absorption between 60 and 90 °C, for all samples, which is indicative of a conformational change. The jump at the ferroelectric transition as PMMA is added to the blend, which indicates the decrease of sample crystallinity, should also be noted. The changes in these absorption bands are completely reversible with temperature: on cooling, the ab-

Table I. Infrared Active Modes for PVDF and P(VDF-TrFE) Polymers in the Crystallographic β Phase[a]

Obs. PVDF (cm^{-1})	Obs. Copo. (cm^{-1})	Calcul. Freq. (cm^{-1})	Attribution[21,24]	Symmetry
3014	3012	3029	ν_a (CH_2)	B_2
2977	2977	2980	ν_s (CH_2)	A_1
	1453	(1456)	$[\delta$ (CH_2) in TG^+TG^- defect]	—
1430	1428	1423	δ (CH_2)	A_1
1400	1402	1396	w (CH_2)	B_1
	1345	1334	$[\nu_s$ (CC) in TG^+TG^- segment]	—
1273	1291	1286	ν_s (CC)	A_1
1180		1182	ν_a (CF_2)	B_2
1071		1065	ν_a (CC)	B_1
983		Inactive	t (CH_2)	A_2
880	886	879	r (CF_2) or ν_s (CF_2)	B_2 or A_1
840	851	825	ν_s (CF_2) or r (CF_2)	A_1 or B_2
	780	(784)	$[\nu_s$ (CF_2) in TG^+TG^- defect]	—
	616	(621)	$[w$ (CF_2) in TG^+TG^- defect]	—
	571	—	(defect)	—
511	506	508	δ (CF_2)	A_1
477	472	470	w (CF_2)	B_1
440	440	443	r (CF_2)	B_2
	413	(423)	$[r$ (CF_2) in TG^+TG^- defect]	—
372	372	—	Head-to-head defect	—
350	350	Inactive	t (CF_2) after ref. 22	A_2
262	—	Inactive	t (CF_2) after ref. 23 and 24	A_2
70	—	72	Lattice libration	B_2

[a] The symbols ν_a and ν_s represent, respectively, antisymmetric and symmetric stretching modes. δ, r, w, and t represent bending, rocking, wagging, and twisting modes, respectively. The calculated values into parentheses correspond to predicted modes in the TG^+TG^- conformation, which are indeed observed in α-PVDF. The data for PVDF are from refs. 21, 22, and 24. For the copolymer, the data are from refs. 23 and 24 and this work.

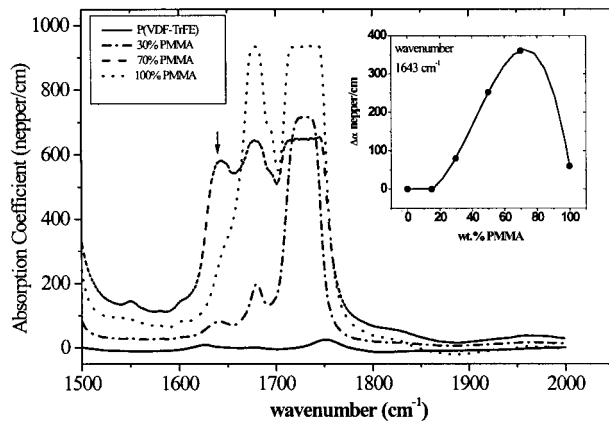sorption increases to approximately the same values (neglecting changes in the crystallinity). Also, no thermal hysteresis has been observed, in agreement with previous measurements with other techniques.[6,7]

The thermal behavior of the absorption band at 616 cm^{-1} is shown in Figure 5, for the samples with 0 and 30 wt % of PMMA. This band, which is normally observed in α-PVDF samples, should be
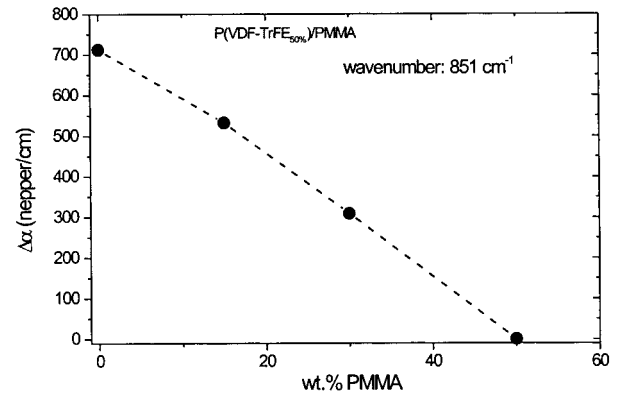

Figure 2. FTIR spectra focusing the 1500–2000 cm^{-1} spectral region, for the same samples of Figure 1. The insert shows the variation of the absorption coefficient of the band at 1643 cm^{-1} as a function of the PMMA content in the blends (the contribution of the absorption band at 1679 cm^{-1} has been subtracted).

Figure 3. Variation of the absorption coefficient of the 851 cm^{-1} band with the PMMA wt % for blends of P(VDF-TrFE$_{50\%}$)/PMMA.
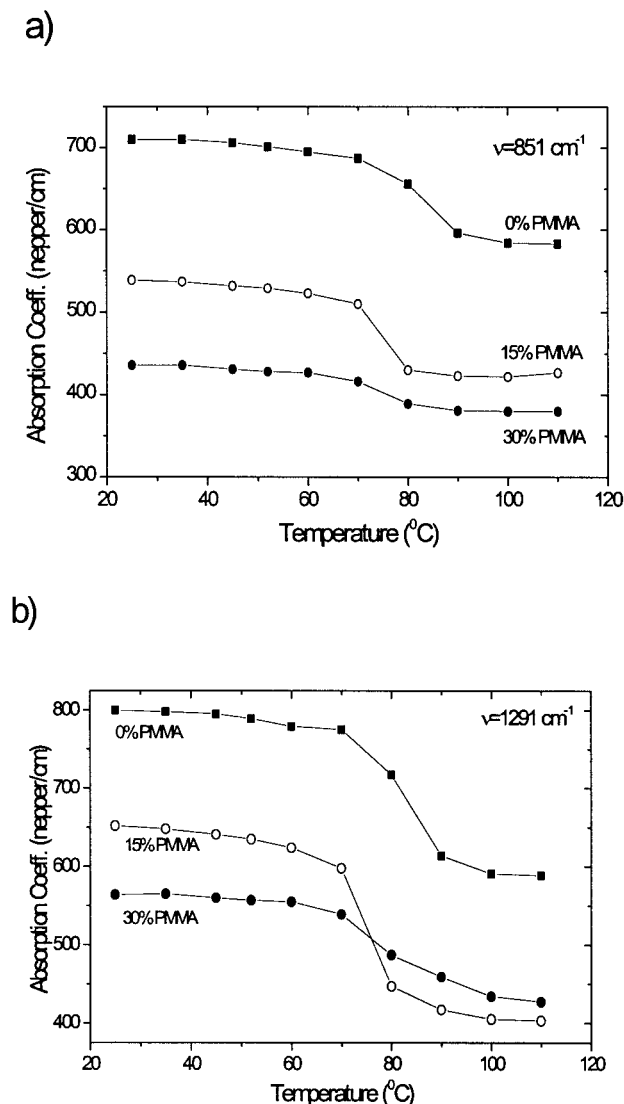
a)

b)

Figure 4. Thermal behavior of the 851 (a) and 1291 (b) cm^{-1} absorption bands for P(VDF-TrFE$_{50\%}$)/PMMA blends with the wt % of PMMA indicated.

due to the presence of TG^{+}TG^{-} defects in the TT chains of the copolymer. Note that the temperature behavior of this band is opposite to those of the TT bands shown in Figure 4 (a) and 4(b), that is, the band at 616 cm^{-1} intensifies just above 60 °C. The decrease of the overall absorption intensity, as the PMMA contents increase in the blend, should also be noted.

Besides the temperature changes in the absorption coefficients for the above-mentioned bands, we also observe that the CH$_2$ stretching modes around 3000 cm^{-1} give clear indication of the phase transition, by shifting approximately 10 cm^{-1} around the transition (the frequencies are smaller in the high-temperature phase).

DISCUSSION

P(VDF-TrFE) copolymers undergo a ferroelectric-to-paraelectric phase transition, where the chains into the crystallites change from a zig-zag (TT) *all trans* conformation (in the polar m2m structure) to a disordered one (helical conformation, in the centrosymmetric 6/mmm group).[13] The mixing with compatible polymers, as PMMA, may affect the ferroelectric and other properties of the copolymer. Saito et al.[5] have conducted measurements of the piezoelectric strain coefficients ($d_{31} = d_{32}$), showing that they vanish when the PMMA contents in the blends exceeds 40 wt %. According to our room temperature X-ray measurements, below 40 wt % of PMMA the system is semicrystalline, with a reduction in the crystallite dimensions along the a and b axes from 100 Å (pure copolymer) to 33 Å (35% PMMA). Above 40 wt % of PMMA, the system becomes amorphous—as inferred from DSC measurements, the ferroelectric and melting transitions disappear above 40 wt % of PMMA[6]. In a recent work,[7] we have confirmed the system amorphization above this value, using dielectric relaxation measurements.

In Figure 1, one can take an overview of the room temperature FTIR spectra of P(VDF-TrFE$_{50\%}$)/PMMA blends for 0, 30, and 70 wt % of PMMA, from 400 to 700 cm^{-1} (a) and from 800 to 4000 cm^{-1} (b). In these frequency ranges, the absorption spectra of blends are just the sum of the individual spectra of the constituents (the same observation holds for the blends with 15, 50, and 100 wt % of PMMA), except for the band at 1643 cm^{-1}, which is active only for the mixture. This interesting result is better presented in Figure 2,
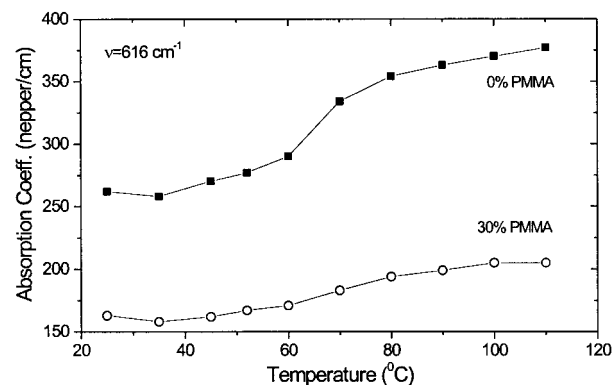

Figure 5. Temperature behavior of the absorption band at 616 cm^{-1} for P(VDF-TrFE$_{50\%}$)/PMMA blends, for samples with 0 and 30 wt % of PMMA.

which focuses the region around this band and gives the evaluation of its corresponding absorption coefficient as a function of the PMMA contents in the blend. This absorption band is clearly an experimental evidence of the strong interaction between the copolymer and the PMMA chains. Note that the band intensity becomes very high when the PMMA content increases from 30 to 50 wt %, indicating that such interaction must reside in the amorphous phase of P(VDF-TrFE), since above 40% the blends are completely amorphous. In order to explain this interaction we may look at the 1730 and 1679 cm^{-1} adjacent absorption bands characteristic of the vibrational modes of the C═O bond of the PMMA side chains (the band at 1679 cm^{-1} may be originated by the stereoirregularity of the PMMA used in our experiments, since it is not present either in isotactic or in syndiotactic PMMA[19]). Although a conclusive attribution to this new band requires additional investigations and calculation by molecular modeling, we suggest that it could be originated from the duplication of the adjacent 1679 cm^{-1} band, caused by the weakening of the C═O bond, which could be sharing its electronic charge with the copolymer chain. In this respect, we note that Coleman et al.[26] and Saito et al.[27] suggested that the carbonyl group could be involved in the interaction between PMMA and PVDF in binary blends of these two constituents. We also remark the similarity between the above result and the 1675 to 1646 cm^{-1} C═O band downshift observed in dimethylformamide/H_3Po_4 gels, which is attributed either to the protonation of the carbonyl group or to the formation of hydrogen bonds.[28]

The 1643 cm^{-1} absorption coefficient of Figure 2 attains its maximum for a sample with high PMMA content (70 wt %). This fact should be explained by the maximum probability to have hydrogen (from the copolymer chains) and carbonyl (from PMMA) with correct positions and orientations to interact.

In order to continue our discussion about the effect of adding PMMA chains to the ferroelectric crystalline phase of P(VDF-TrFE) and the subsequent high temperature spectra analysis, we refer again to Table I, which summarizes the data for the infrared active modes for PVDF and P(VDF-TrFE) in the β phase. Actually, besides some discrepancies between mode assignments, other differences between predicted and observed vibrational bands of the copolymer are normally attributed to the influence of the sample preparation method.[29] The effect of adding PMMA to the

blend can be observed in Figure 3 for the absorption band at 851 cm^{-1}, attributed either to the r(CF$_2$) or ν_s(CF$_2$) mode in an *all trans* conformation of the copolymer (refs. 23 and 24, respectively). As PMMA is added, the intensity of this band decreases and vanishes between 30 and 50 wt % contents. These data are very well correlated with our previous X-ray and DSC results[6]—the disappearance of this band corresponds to the blend amorphization when the amount of PMMA exceeds 40 wt %.

The effect of thermal heating on the blend spectra has been summarized in Figures 4 and 5, which show the behavior of some TT and TG chains during the ferroelectric phase transition for some semicrystalline blends, that is, those which may undergo structural transition. Figure 4 (a) shows the decrease of the absorption coefficient of the band at 851 cm^{-1} for the pure copolymer between 60 °C and 90 °C. As mentioned before, this behavior is characteristic of the ferroelectric phase transition of the P(VDF-TrFE) crystallites that occurs in the above temperature range, according to DSC measurements. The phase transition can also be observed for the blends with 15 and 30 wt % PMMA, although with lower intensity caused by the reduction in the number and size of crystallites. It is interesting to note that the behavior of this band with temperature [Fig. 4 (a)] and with PMMA contents (Fig. 3) is very similar (in both cases the bands tend to vanish), leading to the conclusion that, when the blends become amorphous (PMMA contents higher than 40%), the P(VDF-TrFE) chains assume a rich *gauche* conformation. In Figure 4(b) we can observe the same behavior for the band at 1291 cm^{-1}, which is attributed to ν_s mode of the C—C bond into the TT conformation (see Table I). The invariance of the Curie temperature from 0 to 30 wt % PMMA shows that the ferroelectric structure remains relatively stable, the loose of crystallinity occurring by the reduction in number and size of crystallites.

Figure 5 exemplifies the effect of the ferroelectric phase transition on the TG$^+$TG$^-$ defects in the main TT chains, by focusing in the absorption band at 613 cm^{-1}. During the ferroelectric transition, the corresponding absorption coefficient increases, even for the sample with 30 wt % of PMMA, meaning that the *all trans* segments are changing to rich *gauche* segments during the phase transition.

CONCLUSION

FTIR spectroscopic study of P(VDF-TrFE)/PMMA blends has been performed over a large range of composition, for the copolymer with 50 mol % of trifluorethylene. The spectra revealed an absorption band at 1643 cm^{-1}, characteristic of the mixture and absent in the individual polymers. This band could indicate that the carbonyl group of PMMA would be involved in the interaction between the two components. This interaction would occur only with the amorphous part of the ferroelectric P(VDF-TrFE) copolymer. For PMMA contents higher than 40 wt % the system becomes amorphous and the spectra revealed the disappearance of bands characteristic of the *all trans* conformation. The effect of temperature on the spectra was found to be relevant only for the semicrystalline samples, which present ferroelectric phase transition. The spectra of the paraelectric phase of the copolymer present some similarities with those of its amorphous phase, like the disappearance of the TT dependent bands and the intensification of bands linked to *gauche* segments.

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