Research Article

Effects of Chlorinated Polypropylene on the Conformation of Polypropylene in Polypropylene/Chlorinated Polypropylene/Polyaniline Composites

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We investigated the changes in the conformation and crystalline structure of polypropylene (PP) using a combination of Fourier transform infrared spectroscopy (FTIR), wide-angle X-ray diffraction (WAXD), and differential scanning calorimetry (DSC) based on PP/chlorinated PP (CPP)/polyaniline (PANI) composites. The DSC heating thermograms and WAXD patterns of the PP/CPP/PANI composites showed that the β -crystal was affected greatly by the CPP content. Characterization of the specific regularity in the infrared band variation showed that the conformational orders of the helical sequences in PP exhibited major changes that depended on the CPP content. Initially, the intensity ratio of A_{840}/A_{810} increased with the CPP concentration and reached its maximum level when the CPP content was <13.22% before decreasing as the CPP content increased further. The effect of increased temperature on the conformation of PP was studied by in situ FTIR. Initially, the intensity ratio of A_{999}/A_{973} decreased slowly with increasing the temperature up to 105°C before decreasing sharply with further increases in temperature and then decreasing slowly again when the temperature was higher than 128°C.

1. Introduction

Conductive polymers are expected to yield attractive combinations of properties and are getting more and more attention for researchers from all over the world. Among conductive polymers, polyaniline has the best potential to become economically competitive, for its cheap materials, simple synthetic method, and stability in environment [1]. Although polyaniline has many merits, it is very difficult to be processed in usual methods used in conventional polymers due to its strong intermolecular and intramolecular interactions [2]. For these reasons, wide scale industrial application of polyaniline has been strongly impeded over several years. Many new protonating agents such as sulfonic acids, phosphoric acid esters [2, 3], and phosphoric acid [4] have been introduced in recent years in order to improve the processability of polyaniline. To be worthy and mentioned, it was Cao et al. [5] that firstly introduced sulfonic acid as a protonating agent, and this made polyaniline possible to be processed in solvent. Another method to improve

the processability of polyaniline was that polyaniline was blended with thermoplastic polymers due to polyaniline poor mechanical properties [4, 6–8]. A unique specialty of this method lies in the combination of electric properties of polyaniline and mechanical properties of thermoplastic polymers.

In a series of previous studies, we have prepared PP/CPP/PANI composites by melt processing, and investigated the influence of the concentration of CPP on the electric property of PP/CPP/PANI composites and the influence of CPP on the formation of the intermolecular and innermolecular hydrogen bond in the PP/CPP/PANI composites which is carefully proved by FTIR. We found that the hydrogen bond between CPP and PANI plays a prominent role in the decision of composites' electric property [9, 10]. Does there exist a kind of interaction between CPP and PP in the PP/CPP/PANI composites? Hence, how to detect and identify the interaction between CPP and PP in the PP/CPP/PANI composites becomes one of the most important work.

Since Natta et al. [11] first synthesized high molecular weight isotactic polypropylene (iPP) in 1955, the IR spectroscopy has been used to elucidate the structure of this crystalline polymer. In the infrared spectra of isotactic polypropylene, some absorption bands are connected with the intramolecular vibration coupling within a single chain, and this kind of band is defined as a regularity band or helix band [12, 13]. Therefore, FTIR spectroscopy can provide much meaningful structural information and it is an effective method of denoting the changes of helical conformation of PP [14, 15]. During the past decades, it has been well established that specific regularity bands are related to the different critical length " n " of isotactic sequences [16–18]. For example, the minimum n values for appearance of bands at 973, 998, 841, and 1220 cm^{-1} are 5, 10, 12, and 14 monomer units in helical sequences, respectively. Zhu et al. [17] adopted in situ FTIR and arranged the various regularity bands in terms of the order degree from high to low: 940, 1220, 1167, 1303, 1330, 841, 998, 900, 808, 1100, and 973 cm^{-1} . Hence, the information of effects of CPP on the conformation of PP can be obtained by calculating the absorbance ratios of different regularity bands in FTIR spectra. It has been reported that, for iPP samples when the fully isotactic sequences are very short, iPP crystallizes in the γ form, whereas very long regular isotactic sequences generally crystallize only in the α form [19–23]. Therefore, the crystal structure is greatly affected by the helical conformation regularity.

In this investigation, DSC, WAXD and FTIR are adopted to detect and identify the interaction between CPP and PP in the PP/CPP/PANI composites. The variation of conformation and crystalline structure of PP has been discussed. Conductive polymers are expected to yield attractive combinations of properties, and they are gaining increasing attention from researchers throughout the world. In particular, polyaniline (PANI) is considered to have the highest potential to become an economically competitive conductive polymer due to the cheap materials required for its production, simple synthetic method, and environmental stability [1]. Although PANI has many merits, it is very difficult to process with the standard methods used for conventional polymers because of its strong intermolecular and intramolecular interactions [2]. Thus, large-scale industrial applications of PANI have been hindered for several years. However, many new protonating agents such as sulfonic acid, phosphoric acid esters [2, 3], and phosphoric acid [4] have been employed in recent years to improve the processability of PANI. It should be mentioned that Cao et al. [5] were the first to introduce sulfonic acid as a protonating agent, thereby allowing PANI to be processed in a solvent. Another method for improving the processability of PANI is blending with thermoplastic polymers, which due to the poor mechanical properties of PANI [4, 6–8]. A unique feature of this method is the combination of the electric properties of PANI and mechanical properties of thermoplastic polymers.

In a series of previous studies, we prepared polypropylene (PP)/chlorinated PP (CPP)/PANI composites by melt processing and investigated the effects of the CPP concentration on the electric properties of PP/CPP/PANI composites, as well as the influence of CPP on the formation of

intermolecular and intramolecular hydrogen bonds in the PP/CPP/PANI composites, which were carefully examined by Fourier transform infrared spectroscopy (FTIR). We found that the hydrogen bonds between CPP and PANI have a major role in determining the electric properties of composites [9, 10]. However, there may also be important interactions between CPP and PP in PP/CPP/PANI composites. Thus, detecting and identifying the interactions between CPP and PP in PP/CPP/PANI composites has become a major focus of our research.

In this study, we used differential scanning calorimetry (DSC), wide-angle X-ray diffraction (WAXD), and FTIR to detect and identify the interactions between CPP and PP in PP/CPP/PANI composites. We discuss the variations in the conformations and crystalline structure of PP.

2. Experimental

2.1. Materials. PANI protonated with HCl was supplied by Chengdu Organic Chemicals Co., Ltd. (China) and its electrical conductivity was ca 1 S cm^{-1} . PANI was synthesized using the air oxidation method (China patent: CN1626564A). The granularity of PANI-HCl was about 30 μM .

PP (PPH-XD-045, melt flow index = $3.5 \text{ g } 10 \text{ min}^{-1}$) was supplied by PetroChina Co., Ltd. (China).

Dodecylbenzenesulfonic acid (DBSA) was supplied by Kewei Chemicals Co., Ltd. (China) and its content was about 96% (wt).

CPP was supplied by Sichuan Weiye Chemicals Co., Ltd. (China) and its chlorine content was about 31% (wt).

2.2. Preparation of Composites. PANI protonated with HCl was neutralized with 20% (wt) sodium hydroxide (NaOH) aqueous solution for 48 h (the NaOH aqueous solution was in great excess compared with the amount of PANI-HCl) where the pH of the blend (i.e., PANI-HCl and NaOH aqueous solution) was ca 11, which was followed by filtration and washing with deionized water until the pH of percolate was 6–8. The polyemeraldine base ($\text{PANI}_{\text{emer}}$) obtained was dried under vacuum. The purified and dried $\text{PANI}_{\text{emer}}$ was blended with DBSA, where the molar ratio of DBSA relative to $\text{PANI}_{\text{emer}}$ was 0.5. The blend was stirred for 48 h at ambient temperature, thereby obtaining PANI protonated with DBSA (PANI-DBSA).

The PP/CPP/PANI composites were prepared on a two-roll mill. The compositions of the blends are shown in Table 1.

2.3. FTIR Measurement. The specimens used to obtain FTIR measurements were molded into very thin films. Compression molding was performed in the following conditions: preheating at 180°C for 5 min at low pressure and compression for 10 min at 15 MPa at the same temperature, followed by cooling in the mold to ambient temperature with a cooling rate of $30^\circ\text{C min}^{-1}$ at 15 MPa. FTIR analysis was conducted using a Nicolet 170X FTIR spectrometer (Nicolet Co., USA) at a resolution of 2 cm^{-1} . For in situ FTIR, the spectra were obtained at elevated temperatures using a miniature heat controller. The spectrum was collected every 30 s and a heating rate of 5°C min^{-1} was employed. The temperature was

TABLE 1: Formation of PP/PANI composition (in wt %).

Sample code	PP	PANI _{emer}	DBSA	CPP	Theoretical degree of protonation
PP/PANI 1	87.23	4.10	7.79	0.88	0.5
PP/PANI 2	83.70	4.10	7.79	4.41	0.5
PP/PANI 3	79.30	4.10	7.79	8.81	0.5
PP/PANI 4	74.89	4.10	7.79	13.22	0.5
PP/PANI 5	70.49	4.10	7.79	17.62	0.5

monitored using a copper-constantan thermocouple, which was placed directly on the sample. During detection, the specimen was protected by nitrogen.

2.4. DSC. The calorimetric measurements of all samples were obtained using a NETZSCH DSC 204 at a scan rate of $10^{\circ}\text{C min}^{-1}$ under a flowing nitrogen atmosphere, and the sample weight was ca 10 mg.

2.5. Wide-Angle X-Ray Diffraction. WAXD investigations were performed using a DX-2500 SSC diffractometer (China) in the reflection mode. Cu $K\alpha$ radiation was used at 40 kV and 25 mA. The scan step was 0.06° (in 2θ) with a period of 2 s per step. The β -crystal content was deduced from the X-ray data according to standard procedures [24] using the relationship:

$$k = \frac{H_{\beta(300)}}{H_{\beta(300)} + H_{\alpha(110)} + H_{\alpha(040)} + H_{\alpha(130)}}, \quad (1)$$

where $H_{\alpha}(hkl)$ and $H_{\beta}(hkl)$ represent the heights of the (hkl) peak for the α and β phases, respectively.

3. Results and Discussion

3.1. Effect of CPP on the Crystal Structure of PP. Figure 1 shows the DSC heating thermograms for PP/CPP/PANI blends, which were recorded at a heating rate of $10^{\circ}\text{C min}^{-1}$. In order to clearly demonstrate the differences in the DSC curves, only two curves are shown in Figure 1. The two samples produced melting endotherms with characteristic doublet peaks. The peak at the lower temperature is represented as a shoulder of the peak at the higher temperature. The multiple peaks in the DSC curves are attributable to the melting of the two polymorphic forms of PP [25]. In general, the β form melted at a lower temperature and the α form melted at a higher temperature. Figure 1 also shows clearly that the β peak of PP/PANI 4 is more prominent than that of PP/PANI 2. This demonstrates that the β -crystal is affected greatly by the CPP content.

Figure 2 shows the WAXD patterns for the PP/CPP/PANI composites. PP is crystallizable and it has three crystalline phase types: α -crystal, β -crystal, and γ -crystal. In Figure 2, the presence of α -crystal is indicated by the crystalline peaks at ca 14° , 17° , 18.5° , 21° , and 21.8° , while the crystalline peak at ca 16° is attributed to β -crystal [25]. According to the standard procedures [24], the β -PP contents are 12.7% and 27.7% for PP/PANI 1 and PP/PANI 4, respectively, thereby indicating

FIGURE 1: DSC heating thermograms of PP/CPP/PANI blends.

FIGURE 2: WAXD patterns of PP/PANI 1 and PP/PANI 4.

that the CPP content has a major impact on the β -crystal type. These results are consistent with those obtained by DSC.

3.2. Effect of the CPP Content on the Conformation Structure of PP. In general, the regularity bands of PP are related to the different helical lengths “ n ” of the isotactic sequences [15–17]. The intensity of the regularity bands can reflect the quantity of different helical lengths “ n ” in isotactic sequences. Therefore, the conformational ordering of different samples can be determined based on the absorbance ratios of the various regularity bands.

Figure 3 shows the FTIR spectra of the PP/CPP/PANI composites with different CPP contents, which demonstrate that the intensities of different regularity bands (810 , 840 , 973 , and 999 cm^{-1}) changed slightly as the CPP concentration increased. The variations in these regularity bands indicate that the conformation of PP is affected by the CPP concentration. These regularity bands can be divided into two groups ($810/40\text{ cm}^{-1}$ and $973/999\text{ cm}^{-1}$), which we will discuss in detail later.

FIGURE 3: FTIR spectra of the PP/CPP/PANI composites.

FIGURE 4: FTIR spectra of the PP/CPP/PANI composites with different CPP concentrations (780–856 cm^{-1}).

3.2.1. The Region (780–856 cm^{-1}) of the FTIR Spectra.

Figure 4 shows the FTIR spectra in the 780–856 cm^{-1} region for the PP/CPP/PANI composites with different CPP contents. Obviously, there are two Gaussian bands in this region, where the first band is at around 810 cm^{-1} and the second band is at around 840 cm^{-1} for all of the PP/CPP/PANI composites. In general, the intensity of the two bands was closely related to the size of the two critical lengths “ n ” for isotactic sequences of PP. Each FTIR band has its own intensity coefficient and each sample has its own thickness; thus, the intensities of the regular helical conformation bands should be processed in order to determine the effect of the CPP content. In the present study, the variation in the intensity ratio was used to indicate the effect of the CPP content on the two critical lengths “ n ” for isotactic sequences of PP.

The FTIR results for this region are shown in Table 2, where $\text{XS}_{810} = A_{810}/(A_{810} + A_{840})$ and $\text{XS}_{840} = A_{840}/(A_{810} + A_{840})$. The relationship between the A_{840}/A_{810} ratio and the CPP content is shown in Figure 5.

Table 2 and Figure 5 show that the appearances of bands at 840/810 cm^{-1} for the two critical lengths “ n ” of isotactic sequences were affected by the CPP content. The A_{840}/A_{810}

TABLE 2: Curve-fitting results for spectra of the region (780–856 cm^{-1}).

Sample code	First band			Second band		
	$\nu_1 \text{ cm}^{-1}$	A_{810}	XS_{810}	$\nu_2 \text{ cm}^{-1}$	A_{840}	XS_{840}
PP/PANI 1	809.9	1.137	0.546	840.1	0.944	0.454
PP/PANI 2	809.8	0.669	0.453	839.9	0.807	0.547
PP/PANI 3	809.3	1.753	0.421	839.9	2.410	0.579
PP/PANI 4	809.8	0.697	0.362	839.7	1.229	0.638
PP/PANI 5	810.1	0.671	0.458	839.8	0.794	0.542

ν : wavenumber; A: intensity; XS: percent of A.

ratio determined the ratio of the two critical lengths “ n ” of isotactic sequences. The critical length “ n ” of isotactic sequences was larger for the second band than for the first band. Apparently, the order degree was higher when the ratio was larger. As shown in Figure 5, the A_{840}/A_{810} ratio increased initially with the CPP content and it reached a maximum when the CPP content was 13.22%, after which it decreased as the CPP content increased further. Thus, the critical length “ n ” of the isotactic sequences with a band at 840 cm^{-1} corresponded to that with a band at 810 cm^{-1} , which increased with the CPP content up to 13.22% before decreasing as the CPP content increased further. These results indicate that the conformational structures or the order degrees of the helical sequences of PP were affected significantly by the CPP content. This may be attributable to interactions between CPP and the PP matrix.

3.2.2. The Region (950–1020 cm^{-1}) of the FTIR Spectra.

Figure 6 shows the FTIR spectra in the 950–1020 cm^{-1} region for the PP/CPP/PANI composites with different CPP contents, which demonstrates that there are two Gaussian bands in this region, where the first band is at around 973 cm^{-1} and the second band is at around 999 cm^{-1} . The critical length “ n ” values of the isotactic sequences for the appearance of bands at 973 and 999 cm^{-1} were 2–4 and 5–10 monomer units in the helical sequences, respectively [13]. The intensities of the two bands are shown in Table 3.

In Table 3, $\text{XS}_{973} = A_{973}/(A_{973} + A_{999})$ and $\text{XS}_{999} = A_{999}/(A_{973} + A_{999})$. The A_{999}/A_{973} ratio determines the ratio of the two critical lengths “ n ” of isotactic sequences. The relationship between the A_{999}/A_{973} ratio and CPP content is shown in Figure 7.

Table 3 and Figure 7 show the effects of the CPP content on the A_{999}/A_{973} ratio. Figure 7 demonstrates that the A_{999}/A_{973} ratio changed slightly with different CPP contents, which shows that the critical length “ n ” of isotactic sequences for the appearance of a band at 999 cm^{-1} , which corresponded to that for the appearance of a band at 973 cm^{-1} , was affected slightly by the CPP content.

3.3. Effect of Temperature on the Conformation Structure of PP in the PP/CPP/PANI Composite.

The crystallization of PP composites is usually affected significantly by the process temperature. Thus, to investigate the influence of temperature

FIGURE 5: The relation between the ratio of A_{840}/A_{810} and CPP content.

FIGURE 6: FTIR spectra of the PP/CPP/PANI composites with different CPP concentrations ($950\text{--}1020\text{ cm}^{-1}$).

FIGURE 7: The relation between the ratio (A_{999}/A_{973}) and CPP content.

FIGURE 8: FTIR spectra of PP/PANI 4 at different temperatures.

FIGURE 9: FTIR spectra of PP/PANI 4 at different temperatures ($780\text{--}856\text{ cm}^{-1}$).

on the conformation of PP, the PP/PANI 4 sample was analyzed by in situ FTIR.

Figure 8 shows the FTIR spectra of PP/PANI 4 at different temperatures. There are several regularity bands in spectra that changed slightly with the increasing temperature in the composite such as 810 , 840 , 973 , and 999 cm^{-1} , which indicates that the conformation structure of PP is influenced by temperature. Two regions included four regularity bands (810 , 840 , 973 , and 999 cm^{-1}) will be discussed in detail later. Figure 8 shows the FTIR spectra of PP/PANI 4 at different temperatures. Several regularity bands in the spectra changed slightly as the temperature increased, such as 810 , 840 , 973 , and 999 cm^{-1} , which indicates that the conformation of PP is affected by the temperature. Two regions included four regularity bands (810 , 840 , 973 , and 999 cm^{-1}), which will be discussed in detail later.

3.3.1. The Region ($780\text{--}856\text{ cm}^{-1}$) of the FTIR Spectra of PP/PANI 4. Figure 9 shows the FTIR spectra in the $780\text{--}856\text{ cm}^{-1}$ region for PP/PANI 4 at different temperatures, which demonstrates that there are two Gaussian bands at around 810 cm^{-1} and 840 cm^{-1} . Since the intensity of the

FIGURE 10: Intensity ratio (A_{840}/A_{810}) of regularity bands versus temperatures.

TABLE 3: Curve-fitting results for spectra of the region (950–1020 cm^{-1}).

Sample code	First band			Second band		
	$\nu_1 \text{ cm}^{-1}$	A_{973}	XS_{973}	$\nu_2 \text{ cm}^{-1}$	A_{999}	XS_{999}
PP/PANI 1	973.1	1.288	0.392	998.1	1.993	0.608
PP/PANI 2	972.8	1.101	0.424	999.2	1.494	0.576
PP/PANI 3	972.7	3.177	0.412	998.9	4.537	0.588
PP/PANI 4	972.7	1.442	0.408	999.0	2.089	0.592
PP/PANI 5	972.5	1.028	0.426	999.3	1.385	0.574

two bands is related closely to the size of the two critical lengths “ n ” of isotactic sequences of PP, the relative ratio of the intensity of the two bands can be used to represent the helical conformation of PP.

Figure 10 shows the relationship between the intensity ratio of the regularity bands at 810 and 840 cm^{-1} and temperature, which demonstrates that the A_{840}/A_{810} ratio decreased slowly initially as the temperature increased to 105°C, but it decreased sharply as the temperature increased further before decreasing slowly again when the temperature exceeded 125°C. Furthermore, the size of the critical lengths “ n ” of isotactic sequences related to the appearance of a band at 840 cm^{-1} , which corresponded to that for the appearance of a band at 810 cm^{-1} , decreases slowly initially as the temperature increased up to 105°C, but it decreased sharply as the temperature increased further before decreasing slowly again when the temperature exceeded 125°C. These results indicate that the ordered regularity of PP decreased as the temperature increased. This may be attributable to differences in the behavior of the amorphous or partly ordered zones of PP at different temperatures.

3.3.2. The Region (940–1020 cm^{-1}) of the FTIR spectra of PP/PANI 4. Figure 11 shows the FTIR spectra of the 940–1020 cm^{-1} region for PP/PANI 4 at different temperatures, which demonstrates that there are two Gaussian bands at around 973 cm^{-1} and 999 cm^{-1} . Similar to the bands at

FIGURE 11: FTIR spectra of PP/PANI 4 at different temperatures (940–1020 cm^{-1}).

810 cm^{-1} and 840 cm^{-1} , the A_{999}/A_{973} intensity ratio can be used to represent the order degree of the PP composites.

Figure 12 shows the A_{999}/A_{973} intensity ratio versus temperature, which demonstrates that the A_{999}/A_{973} ratio generally decreased as the temperature increased. The A_{999}/A_{973} ratio decreased slowly initially as the temperature increased up to 105°C, but it decreased sharply as the temperature increased further before decreasing slowly again as the temperature exceeded 128°C. The relationship between the conformational ordering of the helical sequences of PP and temperature was determined from the relationship between the A_{999}/A_{973} ratio and temperature. The order degree decreased slowly initially as the temperature increased up to 105°C, but it decreased sharply as the temperature increased further before decreasing slowly again subsequently. This may be attributable to differences in the behavior of the different ordered zones as the temperature increased. These results were consistent with the relationship between the A_{840}/A_{810} intensity ratio and temperature.

4. Conclusions

The analysis of the DSC heating thermograms and WAXD patterns of the PP/CPP/PANI composites showed that the β -crystal was affected greatly by the CPP content. Furthermore, the crystal structure was affected by the regularity of the helical conformation. FTIR was used to investigate the effects of the CPP content and temperature on the conformation of PP in the PP/CPP/PANI composites, which showed that four regularity bands (810, 840, 973, and 999 cm^{-1}) were related to different critical lengths “ n ” of the isotactic sequences of PP. The results obtained in this study suggest the following conclusions.

- The A_{840}/A_{810} intensity ratio increased initially with the CPP concentration and reached its maximum when the CPP content was lower than 13.22% before decreasing as the CPP content increased further. Thus, the A_{999}/A_{973} ratio was affected slightly by the CPP content.

FIGURE 12: The relation between intensity ratio (A_{999}/A_{973}) and temperature.

- (ii) The A_{840}/A_{810} intensity ratio decreased slowly initially as the temperature increased up to 105°C, but it decreased sharply as the temperature increased further before decreasing slowly again when the temperature exceeded 125°C, where the A_{999}/A_{973} intensity ratio was similar to A_{840}/A_{810} .

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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