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Analysis of Structure Transition and Compatibility of PTT/PC Blend without Transesterification[*]

Qing-long Song[a], Hui-ying Wen[a**], Jesper de Claville Christiansen[b], Dong-hong Yu[c], Chun-sheng Chen[a] and Shi-chun Jiang[d**]

[a] *College of Engineering and Technology, Northeast Forestry University, Harbin 150040, China*
[b] *Department of Mechanical and Manufacturing Engineering, Aalborg University, DK-9220, Aalborg, Denmark*
[c] *Department of Biotechnology, Chemistry, and Environmental Engineering, Aalborg University, DK-9220, Aalborg, Denmark*
[d] *School of Materials Science and Engineering, Tianjin University, Tianjin 300072, China*

Abstract Poly(trimethylene terephthalate)/polycarbonate (PTT/PC) blends were prepared by solvent mixing to avoid transesterification during high temperature blending. The influences of compositions on the thermal behavior, crystallization morphology and structure of the blends were studied. FTIR results indicated that there was no COO linking to two phenyl groups on each side chain and DSC results supported no transesterification reaction. DSC curves showed that T_c and T_{mc} increased to maximum range when PC contents were between 7 wt%–15 wt%, however, T_m decreased constantly with the increase of PC contents. It was observed from POM that PTT spherulitic morphology and crystallization kinetics were obviously influenced by the change of PC contents. Structural evolutions during cooling were investigated by SAXS which showed L_c of PTT remained a constant with different PC contents and also fixed during crystallization, nevertheless, it revealed a maximum value of L_{nc} for sample PTT_{93}. It was concluded that PC chains could be permeated into not only amorphous crystallite structure but also amorphous lamellae structure and 7 wt% PC content was supposed to be the "proper" penetration amount into PTT lamellae structure which led to a maximum capacity of amorphous lamellar layer. Fringed-micelle crystal model was adopted to illustrate semi-crystalline physical structures of the blend in two kinds of component aggregation states.

Keywords: Solvent mixing; Transesterification; Lamellae structure; Fringed-micelle.

INTRODUCTION

Polymer blending is an effective way in industry from which desirable properties of all components could be obtained and tuned as needed[1, 2]. Binary polymer blend of poly(trimethylene terephthalate) (PTT) with polycarbonate (PC) is of commercial interest for potential performance combination of impact strength, modulus, heat, resilience and abrasion resistance[3–5]. Most polymers are thermodynamically immiscible, but for the pair of PTT and PC, there is an important fact that both PTT and PC are polyesters and will react in the way of transesterification at high temperatures. Transesterification is a special pheonomenon which is actually the exchange of carbonate groups in two polyesters. This reaction leads to formation of copolymers comprised of mutually interacting groups which results in an enhancement of the blend miscibility. Many researchers had

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[**] Corresponding authors: Hui-ying Wen (温慧颖), E-mail: hywen@nefu.edu.cn
Shi-chun Jiang (蒋世春), E-mail: scjiang@tju.edu.cn

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studied blends of PTT with many other polyesters. As expected, all the studies focused on the influence of transesterification degree on miscibility[6–10], phase behavior[11–14] and mechanical properties[15–17]. It was reported that the increase of temperature and annealing time helped the occurrence of transesterification and the reaction product could serve as a compatibilizer or even as the main species of blends. Under this circumstance, the change of molecular chain aggregation and regular sequence evolvement during crystallization is actually very difficult to discuss.

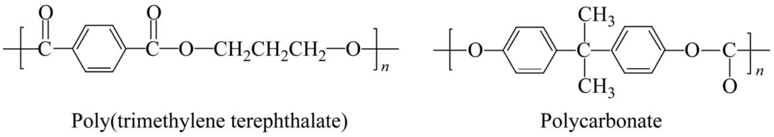
In PTT/PC system, PTT is a semi-crystalline polymer and the crystallization structure is its main aggregation state. Typically such semi-crystalline structure contains well-packed sequence (crystalline) regions with amorphous (liquid-like) chains as disperse phase. Another component PC usually crystallizes to a very small extent for the existence of semi-rigid structure. As we know, the thermal behavior and crystallization morphology are strongly dependent on the crystalline phase structure, whereas, molecular arrangement and aggregation structures of semicrystalline and amorphous phase will influence each other, also affect crystallization kinetics and macroscopic properties of materials which has been reported by many researchers[18–24]. But until now the mechanism how the dynamics and chain motions in amorphous region act on interfaces and affect properties and the final structure of blends is still vague.

In this paper, we aim to prepare PTT/PC mixtures by solution mixing method which could avoid possible transesterification reaction, and the same phase structure inside experimental samples for thermal and crystallization analyses can be processed. The interaction between semicrystalline and amorphous component during crystallization is discussed.

EXPERIMENTAL

Materials

PTT is synthesized by Dupont Chemicals Co with density of 1.31 g/cm^3, and PC is a product from Dow Chemical Co with density of 1.20 g/cm^3. The molecular structures of PTT and PC are provided as follows (Scheme 1):



Scheme 1 The molecular structures of PTT and PC

Before mixing, PTT and PC pellets were dried for 5 h under vacuum at 115 °C to minimize possibility of hydrolysis during heating. For comparative analysis, all samples underwent the same thermally treated history. PTT/PC blends with different compositions were prepared by the method of solution blending with 2:1 volume ratio of phenol and 1,1,2,2-tetrachloroethane as solvents. After dissolving, solvents were evaporated by Rotary Evaporator at 115 °C for 30 min and mixtures were further disposed by vacuum drying before test. Blends with different weight contents of PC were prepared which are abbreviated respectively as PTT$_x$ (x denotes weight percent of PTT in blend, *i.e.*, PTT$_{90}$ means 90 wt% of PTT and 10 wt% of PC).

Characterization

FTIR measurements were performed on a Perkin-Elmer Spectrum One FTIR spectrometer. This spectrometer is configured to work in routine with an attenuated total mono reflection (ATR) device using a diamond crystal with the best resolution of 0.5 cm^{-1}. Universal ATR sampling accessory in ATR modes was used. Mid-IR spectra were measured (4000–6000 cm^{-1}). DSC measurements were carried out on a TA instrument DSC Q2000 and 50-position autosampler, and nitrogen gas was purged at a flow rate of 50 mL/min. Samples (about 6–8 mg) were sealed in aluminum pans and an empty pan was used as a reference. Crystallization morphology observation was performed using an Olympus polarized light microscope fitted with an digital camera and a Linkam CSS450 hot stage. Blends were first heated to 250 °C, then compressed between two quartz plates, remained at melt state for 4 min, after that cooled down at a cooling rate of −30 K/min to 210 °C to proceed isothermal crystallization. Crystallization morphology was recorded by video capture every set intervals. SAXS experiments were performed with the facility at Hamburg Synchrotron Radiation Laboratory (HASYLAB). SAXS data were collected and accumulated in time frames of 12 s. All data were corrected for background and detector response, and then azimuthally averaged for analysis. Finite morphological stack model of crystalline-amorphous structure is usually used to do further analysis where lamellae structural stacks consist of crystalline lamellae layer separated by thin amorphous region, which is characterized by one-dimensional electron intensity correlation function $K(z)$ calculated from Fourier transform of scattered intensity on a relative scale[25].

RESULTS AND DISCUSSION

The blends were evaluated by FTIR and the spectral positions of interest were shown and analyzed in Fig. 1. Carbonyl ($C=O$) stretch band in IR spectrum is deemed to be between 1850–1650 cm^{-1} which depends on specific chemical environments. Carboxyl group in PTT/PC is associated with two characteristic infrared stretching. PC carbonyl stretch peak is at 1768 cm^{-1}, and for PTT the position is at 1709 cm^{-1} because of conjugation function which increases single bond character. Besides carbonyl stretch bond, there are 1502, 1080 and 1015 cm^{-1} which are respectively assigned to PC's ring $\upsilon(c-c)$, $\gamma(c-c-c)$ and $\upsilon_s(O-C-O)$. For PTT there is another characterized stretching vibration of $C-O$ from $-COOC-$ at 1246 cm^{-1}. Peak area ratio of the two kinds of carbonyl characterized bonds is subjected to the component composition in blend. From Fig. 1, with enhancing PC contents the corresponding carboxyl group characteristic peak intensity gradually accumulates.

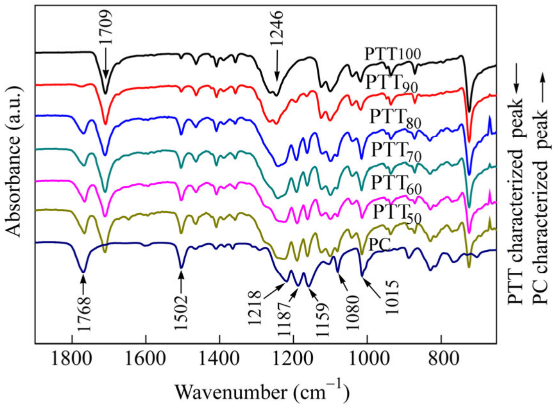


Fig. 1 FTIR spectra of PTT_x

Figure 2 is the amplified part of Fig. 1 in the region from 1900 cm^{-1} to 1625 cm^{-1}, which shows effect of composition on peak positions of the two kinds of $C=O$ stretch mode. Carbonyl characteristic peak at 1709 cm^{-1} which belongs to PTT almost remains constant for PTT-rich blend. With PC content larger than 20 wt%, it shows an slight increase from 1709 cm^{-1} to 1710.5 cm^{-1}. Interestingly, for PC carbonyl stretch mode there shows a very special phenomenon. First what needs to be stressed is that for sample PTT_{80}, PC carbonyl stretch position is the same as pure PC (1768 cm^{-1}). For blends with less PC, it has an obvious shift to higher wavenumber with PC decreasing (*e.g.* for PTT_{98}, almost increased to 1776 cm^{-1}); with PC larger than 20 wt%, it shifts to a lower value (*e.g.* for PTT_{50}, it could reach 1767 cm^{-1}). This kind of shift to higher or lower frequency is attributed to the change of local environments which greatly affected by the composition changing. Generally, strong bond interactions lead to absorption at lower wavenumber because of increasing Force constant-κ in infrared analysis, and vice versa. According to the IR results, the relationship between two kinds of $C=O$ stretch mode influences each other from which we can infer that there is some compatibility at molecular level. To begin with, how the PC chain sequences affect PTT carbonyl surrounding environment is discussed. Three main phases existed in the blend: well-ordered PTT crystalline phase, disordered PTT amorphous phase and PC amorphous phase. When blend is PTT-highly rich, a small quantity of PC chains are deemed to be very scattered which is not enough to change PTT carbonyls interaction and make no difference from PTT carbonyl stretching position. As PC content increased to a certain extent (20 wt%), PC could be acted as a dispersing agent which decrease interactions inside PTT carbonyl bond. On the other hand we pay attention to the influence of PTT chains on PC carbonyl surrounding environment. For a PTT-rich system, PTT can be imagined as a diluent and interaction inside PC carbonyl bond will be weaken for low density distribution. Whereas with the increase of PC contents, there shows a shift of PC carbonyl peak to lower position which bespeaks strong interaction in carbonyl bond. As we know, PC possesses a rigid structure for the existence of benzene rings which make molecular chains very difficult to be neatly arranged. With more PC inside the system, more PC chains will be impelled into limited regions derived from confined space by rejection of growing crystallites during PTT chain segments sequencing, which leads to a intense interaction of PC carbonyl bond. From FTIR results we speculate that there should have intimately mixed entanglements of PTT and PC chains at molecular level and the resulting topological constraints in amorphous regions influenced the final crystalline structure.

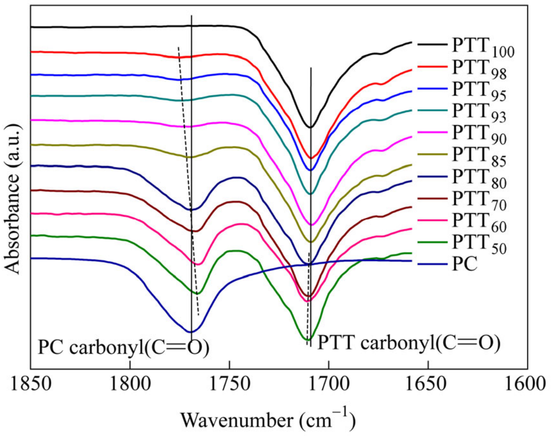


Fig. 2 FTIR spectra of enlarged region of 1600–1850 cm^{-1} generated by two kinds of C=O stretching vibrations

To explore whether transesterification reaction occurred, we made contrasts of series of FTIR and DSC results of PTT$_{80}$. For an efficiently comparative analysis, annealing temperature is set according to experimental melting condition 250 °C and annealing time is chosen up to 1.5 h to initiate transesterification reaction. Figure 3(a) shows that annealing time of 5 and 10 min gives no spectral changes compared to the spectra of the unannealed sample. Once annealing time reached up 15 min, a new band appears at 1070 cm^{-1} which is assigned to a new fully aromatic ester structure, *i.e.*, COO from transesterification product. The new emerging band is more and more observable with longer annealing time. Meanwhile, for samples with more than 15 min annealed history, amplitude of PC carbonyl stretch peak at 1768 cm^{-1} has a non-ignorable decrease, which might be the participation of transesterification reaction. DSC cooling curves for PTT$_{80}$ also drew similar conclusions as shown in Fig. 3(b). Crystallization exothermic peak position and enthalpy with annealing time 5, 10 and 15 min showed no obvious change. With longer melt annealing time applied, a depression of T_c (temperature at exotherm minimum) and enthalpy were observed which attributed to a new copolymer component. The results confirmed that no new copolymer was detected in at least up to 10–15 min melting process which was called "induction period" for transesterification.

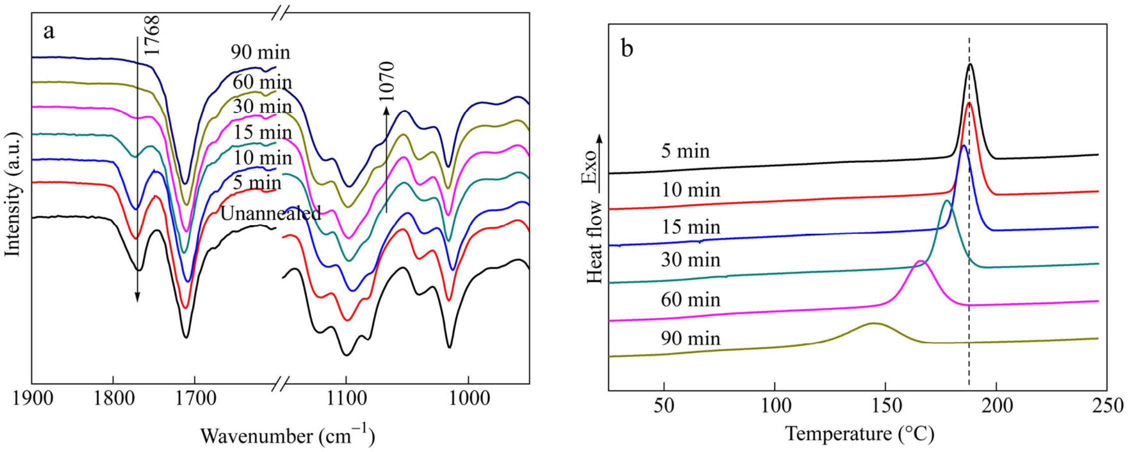


Fig. 3 PTT$_{80}$ (a) FTIR spectra of unannealed and different time annealed at 250 °C and (b) DSC cooling curves at rate of −10 K/min after different time annealing at 250 °C

The blends were subjected to DSC measurements with the following procedures. Firstly PTT$_x$ was heated to 250 °C and kept for 4 min to remove any thermal history, after that it was cooled down to 25 °C at a rapid cooling rate −80 K/min. After being kept at 25 °C for 1 min, it was secondly heated to 250 °C at a rate of 10 K/min and remained for 4 min, then proceeded with second cooling process to 25 °C at a rate of −10 K/min. For the third time, it was reheated to 250 °C at a rate of 10 K/min and finally cooled down to 25 °C. With the supports of FTIR and DSC, transesterification is not considered in our experiments.

Figure 4(a) shows the second heating and cooling scans for pure PTT. During heating there is a clear and sharp exothermic peak at about 197 °C before melting. This high temperature exothermic peak was attributed to melt recrystallization or perfection of defective crystals which were formed at the first rapid cooling round. The heating curve also presented an endothermic peak at 226 °C which was supposed to be melting temperature (T_m). In the cooling process, a well defined DSC endothermic peak at 177 °C (T_c) indicated cooling crystallization process. Figure 4(b) shows heating curves for all blends, and two T_gs are observed at about 50 °C and 120 °C which separately belongs to PTT and PC. A slight enhancement for low temperature T_g with increasing contents of PC (T_g reaches 55 °C for PTT$_{50}$) suggested that PC component molecularly mixed with PTT phase. In the same time, high temperature T_g showed a trend of decreasing with increasing PTT contents. The system exhibited two distinct glass transitions and the two transitions had shifted towards each other, which meaned it was semi-miscible or partially miscible.

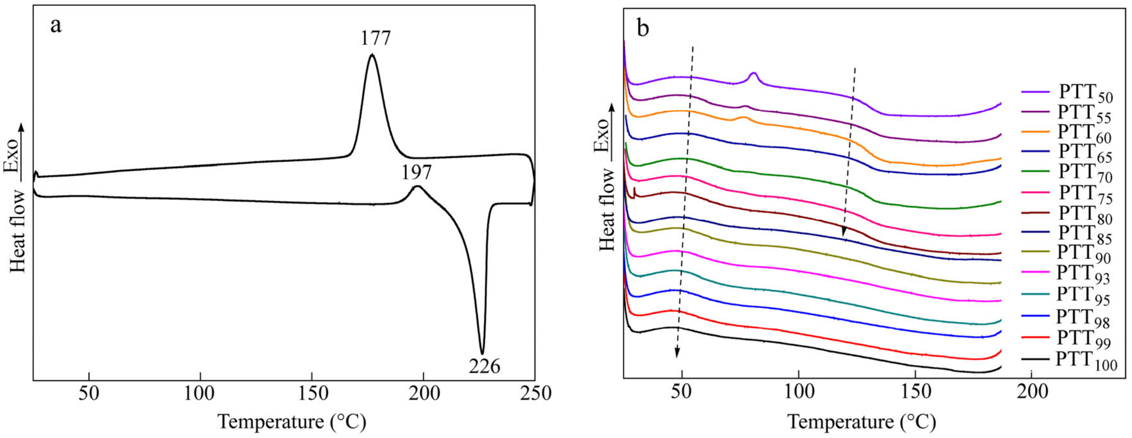


Fig. 4 DSC scans (a) the second heating and the following cooling process for PTT$_{100}$ and (b) the second heating curves for PTT$_x$

Figure 5 shows two cooling and two heating DSC curves from which the influence of the compositions on thermal behavior could be analyzed. Firstly, we focus on cooling curves (as shown in Figs. 5a and 5c). T_c value shows an obvious change which is obviously influenced by compositions. It increased with PC increasing for the PTT-rich system and then decreased after PC content is larger than 15 wt%. There shows a "proper" content of PC which could improve PTT crystallization kinetics in the process of cooling crystallization. With slower cooling rate −10 K/min (Fig. 5c) T_c had the same changing trend and increased T_c and much more-defined endothermic regions were shown. As we know, crystallization of polymers is a process associated with alignment and rearrangement of molecular chains or segments. An ideal state of polymer chains ordering is with an infinitely large sample and an infinite time to reach a complete equilibrium of crystallization according to Flory theory[26]. When polymer crystallizes at a lower cooling rate, there is much more time for molecular (or segmental) movements to reach a more ordered state and hence more perfect crystallization structure can be obtained. In the meantime, the degree of increase of T_c for the PTT-rich system at a higher cooling rate is much more obvious. Figure 5(a) reveals a notable increase of T_c (from 146 °C to 165 °C) with PC adding up to 7 wt%, by contrast, Fig. 5(c) shows a relatively less increase (from 177 °C to 187 °C) which means the higher cooling rate increases the gap of crystallization equilibrium time. With PC content over 15 wt%, the increasing trend of T_c goes into sharp reverse which is very worth discussing.

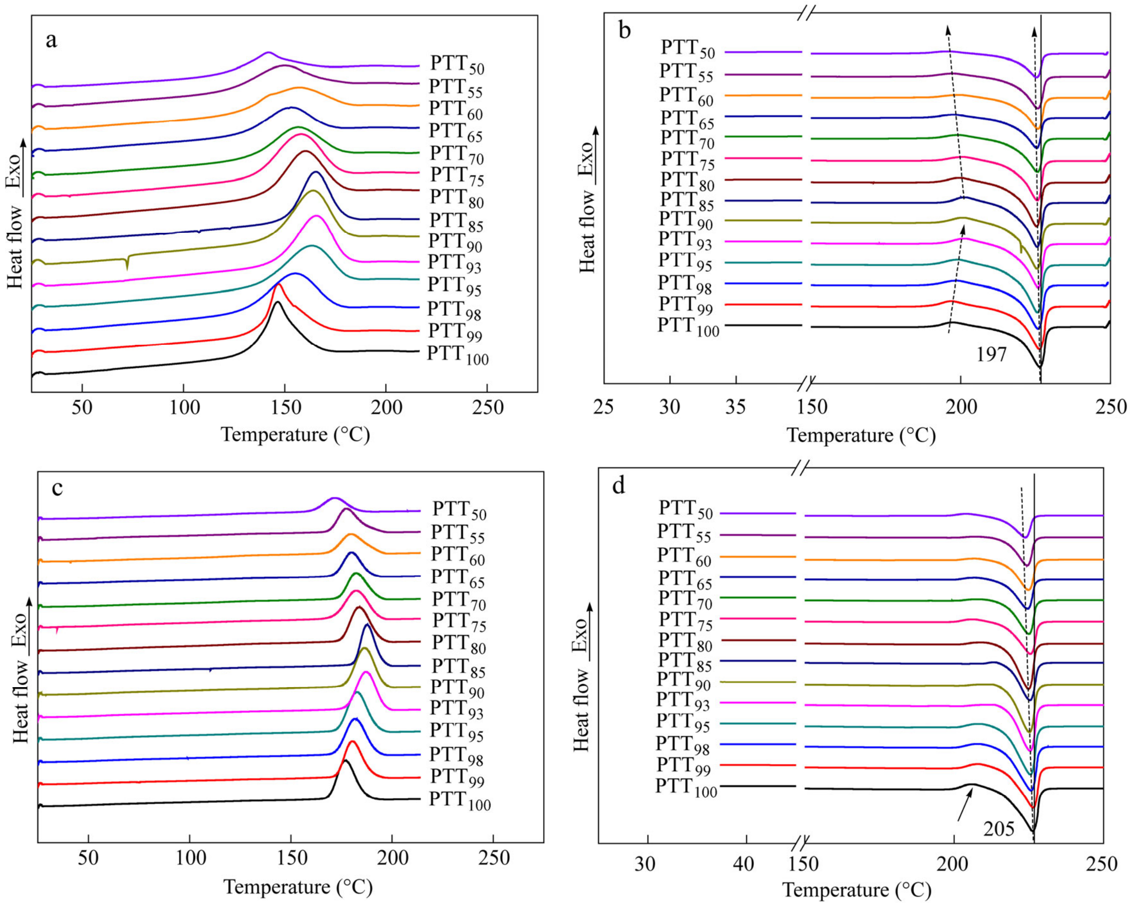


Fig. 5 DSC curves of PTT/PC blends with different PC content during (a) the first cooling process at cooling rate of −80 K/min, (b) the second heating process at heating rate of 10 K/min, (c) the second cooling process at cooling rate of −10 K/min and (d) the third heating process at heating rate of 10 K/min

It is apparent from FTIR and DSC that amorphous PC has a pronounced influence on the ordering of PTT crystalline phase, but few people discuss its influence mechanism. In melt state, long chains are a disorganized and in entangled state. Chain entanglement was typically regarded as a special type of intermolecular interaction, which would hinder the large-scale motions of chains, prevent to align parallel to each other and achieve a regular conformation, and finally a less completed ordered crystal structure was obtained[27, 28]. Hoffman reported that the entanglement points could act as obstacles or additional to friction in the reptation of polymer chains which had an effect on primary nucleation of crystals and their growth rate[29]. During crystallization, PTT chain segments are sequenced forming lattice inside crystalline phase, at the same time some PTT molecular chains are disordered and have an incomplete disentanglement. Beyond that, there is the third phase-PC amorphous phase existing in the blend. Some PC entanglement points could be involved in PTT phase structure making sequence ordering process more complicated. Under the circumstance, we can speculate that the integrity and ordering of crystalline sequence would be always affected by entanglements from both PTT chains and PC chains in the blends. In the PTT-rich system, rigid PC chain structure could be served as blocks for formation of entanglement points of PTT chains which somehow increases fluidity of the crystallizable phase and helps ordering of sequence chains arrangement. Besides, the special rigid PC chains could be acted as an effective heterogeneous nucleating point which will improve the crystallization dynamic behaviors of PTT. The two factors are the main reasons why T_c will increase with the enhancing amount of PC within a certain limit. Results showed with more PC component (> 15 wt%) the progress of PTT sequence ordering was impeded for changed crystallization circumstances and blend systems had to pursue lower thermodynamics for crystallization. Under experimental conditions, more rigid PC chains will induce the increase of confined space for intact ordered PTT long chain sequences, which creates difficulty in the formation of effective crystallization sequence structure during cooling and finally the system has to seek for proper thermodynamics condition at lower temperatures.

The second and third heating curves of DSC (Figs. 5b and 5d) show that all blends exhibit obvious melt-recrystallization or as-called second crystallization process. As we discussed above, there was an "proper" composition of the blend which could help undergo a highest thermodynamics level during crystallization. This phenomenon was also observed in melt-recrystallization process which showed that for blend with "proper" PC content (15 wt%) melt-recrystallization temperature (T_{mc}) was highest. As marked by dash line in Figs. 5(b) and 5(d), with PC content increasing T_{mc} shifted to higher value and later had a reverse shift. With slower cooling history (Fig. 5d), molecular chains had longer time to arrange and assemble into more ordered structures which would result in a relatively higher T_{mc}. In the initial period of melting, defective crystals had their chance for perfection and some short polymer chains could proceed readjustment and make a more ordered arrangement. Recrystallization resulted in similar perfection of final crystalline structures in blends, which was responsible for the fact that T_m showed no big difference for all blends. T_m shifted slightly to lower temperature as marked by the dash line with PC increasing and was accompanied by decreasing endothermic enthalpy, which meaned that blend with high PC content exhibited less perfect crystalline structure and volume crystals. In general, we found that an "proper" composition range (PC contents were between 7 wt%–15 wt%) accelerated thermodynamics. However, the perfection of crystalline structure constantly decreased with increasing PC. From the FTIR and DSC results we speculated that PC chains might have accelerated nucleation process and in the meantime hindered growth and perfection of PTT crystalline. The two effects influenced and restricted each other which directly resulted in a complicated sequence structure during crystallization.

PTT crystalline morphology in blends was observed by POM, as shown in Fig. 6. POM was monitored showing development of crystal morphology during 210 °C isothermal crystallization process. Crystallization morphology is determined by crystal developing environment concerning an interfacial structure and external growth of ordered chain sequence. It is shown that pure PTT displays smooth and perfect concentric ring-banded spherulites. With PC increasing the texture of PTT spherulites is fuzzier and coarser, and the edge of spherulites becomes open with less well-defined boundaries. Meantime, the size of PTT sphere crystals with more PC content is smaller even after the longer crystallization period, which indicates the growth of sphere crystal is obviously delayed by PC's addition. It proves that PC chains act as a crucial factor both in molecular dynamics and microstructure development during PTT crystallization.

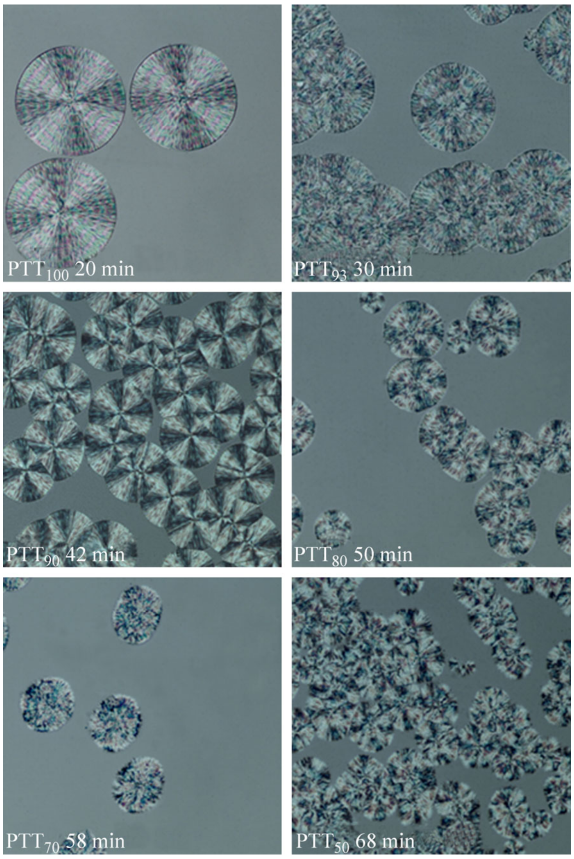


Fig. 6 POM micrographs of PTT$_x$ isothermal crystallization at 210 °C with different crystallization time

To further investigate the influence of PC on the PTT lamellar structure, SAXS data for PTT$_x$ during cooling crystallization (cooling rate −50 K/min from melt) were recorded. Long period (L) and lamellae thickness (L_c) are shown in Fig. 7 which derived from self-correlation triangle in the $K(z)$ curve calculated from Fourier transform of scattered intensity[25]. We noticed that L_c remained almost unchanged (3 nm) in the process of cooling and it also did not change with varied PC content. We assigned 3 nm to L_c rather than amorphous region thickness L_{nc} under the condition that rapid cooling condition made it hard to achieve high crystallinity degree with the support of DSC result. During cooling crystallization, ordered lamellar structure keeps perfecting and some new lamellae will be inserted between the already-existing ones which cause the decreasing of L and give the final value around 8 nm under experimentally observable range. It is worth noticing that L has increased in PTT/PC blends and there shows a maximum thickness value of L_{nc} for PTT$_{93}$. It seems that PC polymer chains not only can be permeated into amorphous region but also can be inserted into PTT lamellae structure which causes "capacity expansion" in lamellae amorphous phase structure. For PTT$_{93}$ PC chains may reach a maximum degree of penetration into amorphous region which is corresponding to the tendency of DSC and FTIR results. According to the SAXS data more PC in blend are deemed to more confined space inside amorphous region which may cause chain segments harder to stretch and there should be a spontaneous phase separation between PC phase and PTT amorphous phase. Under the circumstance it is more difficult for PC chains to further affect lamellae amorphous layer structure.

We noticed that L_c for all blends was about 3 nm which was almost the same size of melt crystallized PTT as reported in many results[30−33]. There are two models—chain folding model and fringed-micelle model which all have been commonly used to explain the formation of crystallite structure[34−37]. In the PTT/PC blend L_c is about 3 nm, whereas *c*-axis (chain axis) of required unit cell in PTT is almost between 1.8−1.9 nm which makes

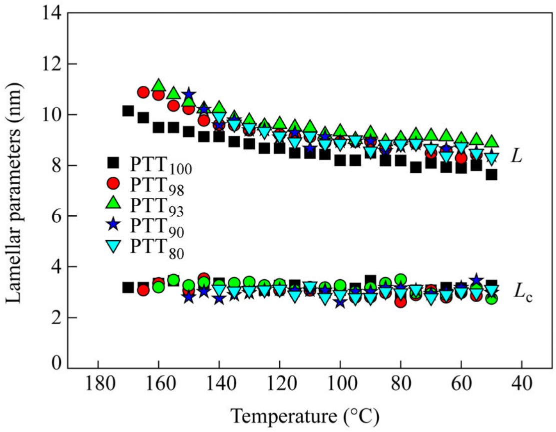


Fig. 7 The evolutions of L and L_c for PTT_x non-isothermal crystallized during cooling as function of temperature

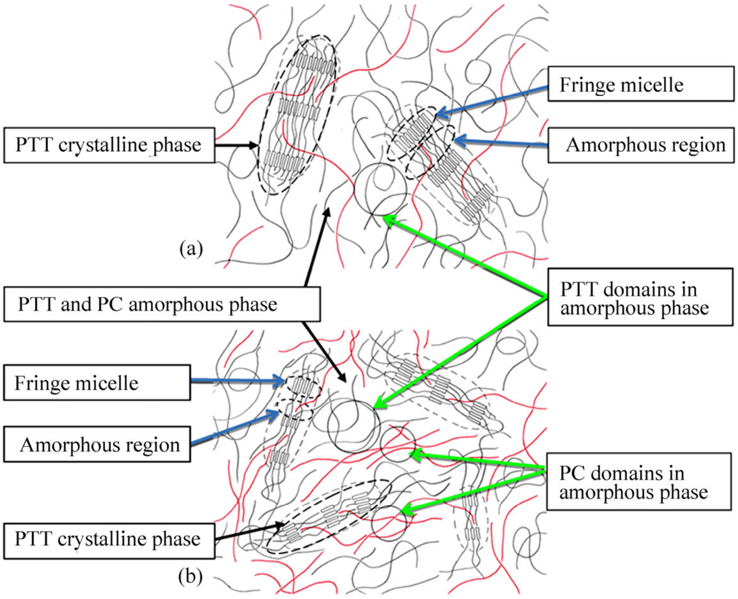


Fig. 8 The schematic illustration of aggregation state (PC chains are marked in red and PTT chains and sequence segments are represented by blue lines) in (a) monophase system and (b) multiphase system

it impossible to fulfill chain folding to achieve phase transformation to edge-on crystalline packing. In this paper, fringed-micelle crystal model is adopted to present the stacking of molecular segments in crystalline region and only under such condition the size of fringe micelles along chain direction can be related to the nanometer scale of stacking periodicity resulted from SAXS analyses. While chain folding is not involved, successive participation of individual PTT molecular stems is not likely to result in efficient prevalence of lamellar organization. A schematic illustration of PC polymer chain aggregation state and stacking of PTT sequence segments in two different aggregation states is shown in Fig. 8. Long molecular chains run through several fringe micelles and the growth of edge-on lamellar stacking is along molecular chain from anisotropic platelets morphology observation, which makes these micelles link with each other and surround by disordered regions. Accompanying with crystallization of PTT, micelles are associated and epitaxially grown on the surfaces of PTT crystals. For the PTT-rich blend (monophase system shown in Fig. 8a), PC chains mix with PTT chains sufficiently. When adjacent PTT chains come together with parallel orientation to form a fringed-micelle/or bundle-like crystallite, some PTT and PC chains are diffused away from the front of PTT crystallization at a rate which is not sufficient to let them completely move away. Some PC chains are just be stuck in lamellae amorphous layer, which is the essential reason that PC shows a "insertion" state inside crystalline region. Under the circumstances, PC chains are very scattered inside PTT chain structure and PTT can be imagined as a diluent for PC molecular chains as we discussed above. With increasing PC in blends (Fig. 8b) more rigid structures exist in system which will correspondingly cause more confined space inside amorphous region. Especially accompanied by PTT crystallization, the rigid PC chains will be impelled into a limited region where PC segments are even harder to stretch or flow. In this case, the system is transformed from monophase to multiphase which comprises amorphous-amorphous interphase and phase aggregations and the two factors begin to play a major role on crystallization morphology and structure of PTT_x.

CONCLUSIONS

Thermal behavior, crystallization morphology and structural evolution of solvent-mixed PTT/PC blends without transesterification reaction were explored by FTIR, DSC, POM and SAXS techniques. DSC indicated that PC content had a crucial influence on thermal behavior. A specific composition range (PC contents between 7 wt%−15 wt%) was deemed to accelerate PTT crystallization kinetics effectivly, whereas, perfection of crystallite constantly decreased with increasing PC contents. It was proposed that PC polymer chains accelerated nucleation process and in the meantime hindered growth and perfection during PTT sequence ordering. It was observed from POM that PC dramatically influenced crystallization kinetics and morphology of PTT spherulites. From SAXS analysis a "proper" PC content 7 wt% was found to lead to an maximum of L_{nc}, whereas, L_{c} almost remained as a constant with varied compositions. All the results verified that PC chains had a crucial influence on the molecular sequence ordering and molecular dynamics of PTT crystallization, hence affected thermal behaviors, crystallization morphology and microstructure of the blend. PC chains affected the crystalline phase structure mainly through the mutual compatibility with PTT amorphous phase and even the insertion into PTT lamellae amorphous layer structure. We deem that it is a monophase system for PTT-rich blend, and with increasing PC the chains are partially miscible in PTT phase structure. Much more PC in blend will induce the generation of confined space which is responsible for less intact ordered PTT long chain sequences and the formation of more defective crystallization structure. In other words, there is a "dividing point" between monophase and multiphase systems when segregated PC segments in the amorphous phase begin to appear.

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