Effects of Sequence Mixing of Stereocomplex Poly(Lactic Acid)/Propylene-Ethylene Copolymer Blends for Plastic Packaging

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Abstract. *This work examines the effect of sequence mixing stereocomplex poly(lactic acid) (Sc-PLA) on the thermal and mechanical properties of Sc-PLA/elastomer blends. In this study, PLA was melted and blended with poly(D-lactic acid) (PDLA) in an internal mixer at the ratios of 95/5, 90/10, and 85/15 by weight, and then the sample was molded by compression molding. The most significant improvement was found in blending 10 wt% PDLA. The results show the highest Young's modulus at 2200 MPa from the neat PLA at 1800 MPa. The stereocomplex PLA/PDLA (Sc-PLA/PDLA) at proportion 90/10 wt% by being chosen blended with propylene-ethylene copolymer (PEC) elastomer to study the effect of sequence mixing of Sc-PLA/PDLA/elastomer blends on thermal and mechanical properties and the amount of PEC elastomer was fixed at 10 wt% in the mixture. The mechanical properties show results at 10% break elongation of Sc-PLA/PDLA/PEC blends that is higher than the others, which confirmed that PEC elastomer, which is improved features of Sc-PLA/PDLA flexibility blends and sequence mixing of PLA stereocomplex/elastomer blends affected to the mechanical properties. The result of thermal properties shows the sequence mixing of PLA stereocomplex/elastomer blends does not affect the melting temperature but affects the enthalpy (ΔH_m-ΔH_c) by Sc-PLA/PEC add PDLA significant decreasing at 23 J/g °C by the other conditions show about 28 J/g °C. It supports the sequence of mixing at Sc-PLA/PDLA/PEC mixed simultaneously, which is the best. The analysis results show that sequence mixing significantly affects polymer blends' properties, which can be applied to industrial processes.*

Keywords:

Poly(Lactic Acid), Stereo complex, Propylene-Ethylene Copolymer.

1. Introduction

The growing use of petroleum-based materials and the production of plastic waste have caused significant environmental problems worldwide. Plastic is low-cost, lightweight, strong, heat-resistant, and flexible. Therefore, plastics are used as a component in many products, including general appliances such as bowls, tables, chairs, and toys, as well as food and beverage packaging that are disposable, such as plastic bags for food, plastic plates, plastic bowls, plastic spoons and forks, water bottles, and snack boxes, etc. [1].

Developing bio-based and biodegradable polymers as alternatives to traditional petroleum-based polymers has received increasing attention. Poly(lactic acid) (PLA) is one of the most popular biopolymers owing to it being biodegradable, producible from renewable resources (e.g., corn, cassava, sugarcane), non-poisonous on the human body and the environment, biocompatible and easy processability. As a result, it is used in large-scale applications from biomedical materials to industrial packaging materials. However, PLA has two limitations: its inherent brittleness and low thermal resistance [1].

PLA's thermal resistance might be enhanced by inducing the formation of stereocomplexation [2]. Ikeda et al. [3] found that results of stereocomplex crystals might be formed by solution or melting to blend enantiomeric PLA, poly(L-lactic acid) (PLLA), and poly(D-lactic acid) (PDLA). These stereocomplexation results show a higher melting temperature (~50°C) than homopolymer, and the presence of stereocomplex crystals can improve thermal and mechanical properties such as tensile strength or Young's modulus. In addition, the composition of the two crystallite domains also depends on the blend content, with the stereocomplex content peaking in a 1:1 blend and diminishing as the mixing ratio moves away from the equimolar value [4]. The mechanical properties of PLA might be developed by mixing with elastomers using rubbers [5-9], plasticizers [10], and other elastomers [11-14]. However, mixing PLA stereocomplex with elastomers also affects the crystallization behavior, formation, and dispersion characteristics of stereocomplexation [13].

Propylene–ethylene copolymer (PEC) improved the tensile strength, strain at break, and percent strain at break of PLA blends, leading to improved flexibility and mechanical performance. PEC showed superior results among various elastomers, making it ideal for applications requiring a durable yet flexible material, such as packaging[15]. As a copolymer of PEC, this material is versatile and can improve its performance and fill the disadvantages of bioplastics. Moreover, it complements the disadvantages of bioplastics, as mixing two different monomer units (propylene and ethylene) improves structural and functional properties. In addition, adding PEC can delay the degradation of bioplastics and extend their service life. However, the blending ratio must be designed appropriately for the application [15-17].

This study used propylene-ethylene copolymer (PEC) to improve the elasticity of Sc-PLA blends. The effects of stereocomplex, PEC blending, and mixing sequence were investigated regarding mechanical properties, morphology, crystallinity, and thermal properties. The results of the work are suitable for application in industrial processes.

2. Preparation

2.1 Materials

PLA with a melt flow index of 6.0 g/10 min and specific gravity of 1.24 g/cm^3 under the trade name of “IngeoTM Biopolymer 2003D” was produced by NatureWorks LLC, USA. PDLA with a melt flow index of 20 g/10min and specific gravity of 1.24 g/cm^3 was produced by Luminy D070. PEC with a melt flow rate of 8 g/10 min and a density of 0.867 g/ml was purchased by Chemical Innovation Co., Ltd and the trade name of “NIPOL DN3320,” grade 3300.

2.2 Preparation of PLA/PDLA Blends

PLA and PDLA were dried in an oven before blending with mixture 60ºC for 4 hours and overnight. PLA/PDLA blends were prepared by melt-blending in an internal mixer at 180ºC and using rotor speed of 80 rpm for 15 min. The PDLA proportions were 5, 10 and 15 wt%.

2.3 Preparation of PLAs/Elastomer Blends

PLA and PDLA were pre-dried in an oven at 60°C for 4 hours prior to their inclusion in the mixture and allowed to dry overnight before blending. The PLAs/elastomers blends were prepared using an internal mixer operating at 180°C with a rotor speed of 80 rpm. The optimal proportion of PLA to PDLA was determined to be 90:10, while the type and content of the elastomer were fixed at 10 wt% PEC. The components were mixed in specific weight percentages (wt%) and in predetermined sequences to evaluate the mixing process, which was categorized into three models: (1) Mixer all three components, namely PLA, PDLA and PEC, in a mixed inside. (2) PLA and PDLA were mixed first, then add PEC. Finally, (3) PLA and PEC were mixed first, then added PDLA. The mixing began by feeding the components that must be mixed into an internal mixer for 8 mins, process in 180ºC and 80 rpm, before adding the specified amounts of components that must be mixed into the chamber. The total mixture time on 15 min.

2.4 Polymer Characterization

The Fourier transform infrared (FT-IR) spectroscopy was applied to verify the chemical structures on a Nicolet iS5 spectrometer. The universal testing machine (UTM) model ASTM D 638 EZ Test, EZ-LX/EZ-SX Series, was used to characterize the mechanical properties. Thermal properties were characterized by TGA-DSC (SDT Q600, TA Instruments). X-ray diffraction (XRD) analysis is a unique method for determining crystallinity by XRD (D8 ADVANCE).

3. Results and Discussion

3.1 Mechanical Properties

The mechanical properties of Sc-PLA blends are shown in Table 1. It was found that elongation at break decreased from 6.3, 5.3, and 4.7% in proportion to PDLA, including tensile strength and stress at break, which are increased when making a stereocomplexation in only 5%PDLA. However, the 10%PDLA shows a great result in which Young’s modulus value increased, a non-significant decrease for others. Moreover, H. Tsuji shows that the PDLA that closed with a racemic mixture showed higher stereocomplex crystallization [1]. Therefore, Sc-PLA at 10% PDLA was selected to investigate polymer blend sequencing.

The results show that the sequence of mixing was affected by the properties. All mechanical properties show that the PEC was added in the last step, which shows the best mechanical properties. It can be from the completely stereocomplex crystal forming before blending with the PEC for elastic at the end. It can be seen from the elongation at a break value of 9.7%, as seen from %X_c in Table 3. It will not affect crystal formation but will still increase the break elongation. The lowest properties are shown for the PLA/PEC before adding PDLA. It can be described that the PEC obstructed the stereocomplexation. Also, when added, the materials simultaneously show the properties were mid between the two samples. It can be supported that some parts of PEC inhibit stereocomplexation, and some parts of PDLA can form the crystal formation [2].

Table 1 Summary of comparison of mechanical properties of polymer blends at different composition and sequence mixing.

Sample	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Neat PLA	58.2±2.5	1815.1±78.0	5.3±0.4
Sc-PLA/PDLA (95/5)	61.0±1.6	1920.5±90.1	6.3±0.3
Sc-PLA/PDLA (90/10)	54.1±1.6	2211.6±79.8	5.3±0.5
Sc-PLA/PDLA (85/5)	31.3±9.6	1953.0±126.7	4.7±0.5
Sc-PLA/PDLA/ PEC	44.6±2.8	1595.5±95.4	9.0±0.4
Sc-PLA/PDLA add PEC	50.0±4.5	1720.8±73.2	9.7±0.7
Sc-PLA/PEC add PDLA	36.8±5.0	1664.1±74.4	6.7±0.4

3.2 Chemical Structure

The functional group, chemical structure, and crystal formation were characterized by FT-IR analysis, as shown in Fig. 1(a). The peak at 908 cm^{-1} showed the crystal characteristic of the stereocomplex crystal, and that at 921 cm^{-1} reflected the characteristic of homocrystalline. The peak at 955 cm^{-1} is the CH_3 amorphous phase. It can be seen that all stereocomplexes formed 100% of Sc-crystal because of the non-appeared 921 cm^{-1} band. The difference is the intensity of the stereocomplex, which is higher when added more PDLA into the PLA matrix (normalized by amorphous phase at 955 cm^{-1}), as shown in Fig. 1(b). However, the 90/10 and 85/15 cannot see a significant change[3]. It can be supported to pick up the ratio 90/10 to blend with the PEC because of its higher mechanical properties. The FT-IR spectra of Sc-PLA/PDLA blends in Fig. 1(b) still showed the band at 1750 cm^{-1}, which the C=O described the strength of bonding and arrangement of crystals in the blends. Other typical bands of C=O also slightly shifted to a lower wavenumber region when increasing PDLA contents. It can be indicated that C=O was stronger with increasing PDLA contents [18, 19].

After that, the PEC elastomer added in the Sc-PLA/PDLA (90/10) blends, the band of C=O would be slightly shifted to a higher wavenumber, it can be indicated that PEC elastomer decreased the strength of C=O of Sc-PLA/PDLA blends. The results showed that the crystal arrangement of Sc-PLA/PDLA (90/10) was better than Sc-PLA/PDLA/PEC blends in every sequence. In addition, the peak area normalization of the crystals by calculating the peak area at 908 and 955 cm^{-1}. The result showed that the number of stereocomplex crystals of Sc-PLA/PDLA/PEC blends was not significantly different between mixing sequences [20].

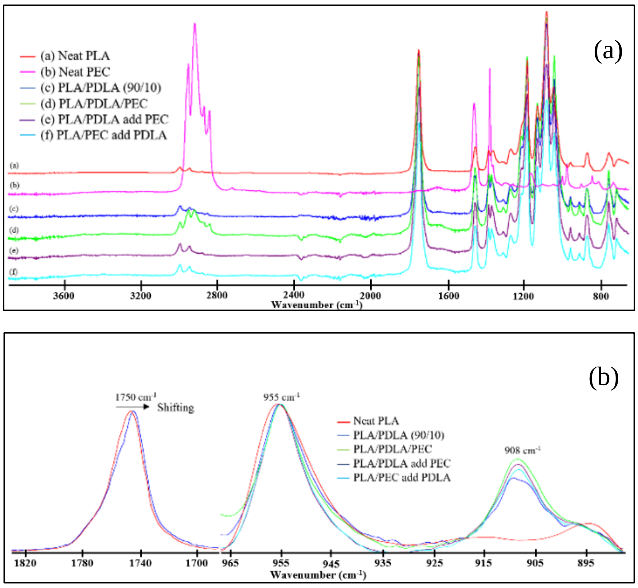

Fig. 1 FT-IR spectrum of polymer blends at different composition and sequence mixing

3.3 Thermal Properties

Thermal properties of neat PLA, Sc-PLA/PDLA, and Sc-PLA/PDLA/PEC blends were investigated by DSC in terms of glass transition temperature (T_g) and melting temperature (T_m) of pure PLA and Sc-PLA/PDLA blends are shown in Table 2. Tg of all samples were quite similar, but Tm was different. The results showed that the melting temperature of PLA was at 150 ºC and slightly decreased when increasing the PDLA content. However, when PLA blended with PDLA, stereocomplexation was formed. The melting temperature of the stereocomplex increased to approximately 210 ºC due to their stronger crystalline structures than the homocrystalline.

In the stereocomplex, two split peaks of ΔH_m are shown. It can describe the enthalpy of PLA and Sc-PLA/PDLA blends by $\Delta H_{m,hc}$ showed the enthalpy at 150 ºC, and $\Delta H_{m,sc}$ showed the enthalpy at 210ºC, as shown in Table 2. The results showed that the value of ΔH_m-ΔH_c will increase with increasing the PDLA content. The results could be that the effect on thermal properties came from the percentage crystallinity of the stereocomplex because of the value of ΔH_m-ΔH_c, which showed an increasing amount of crystallinity of the sample. As a result, corresponding with FT-IR analysis showed a higher peak area of stereocomplex crystallites and a lower peak area of PLA homopolymer in Sc-PLA/PDLA blends with increasing PDLA content, respectively. In addition, the result corresponding with XRD analysis showed that the degree of crystallinity increases as PDLA contents in the Sc-PLA/PDLA blends increase. However, the melting temperature of homopolymer still appeared because all blends consisted of homopolymer and stereocomplex together, which occurred. After all, the ratio of Sc-PLA/PDLA was not 1:1. Regarding PEC blended, the melting temperature and enthalpy show the similarity of different sequences. It can be described that the PEC was not affected by the co-crystalline of the polymers. As a result, it corresponds with FT-IR analysis that showed similar peak area trends of stereocomplex crystallites at different sequence mixing [21, 22].

The thermal stability of neat PLA, Sc-PLA/PDLA, and Sc-PLA/PDLA/PEC blends were analyzed using TGA. Each sample's thermal decomposition (% weight loss) was measured as a function of temperature. The temperatures corresponding to specific weight losses, such as 5% (T_{d5}), 10% (Td_{10}), and 50% (T_{d50}), were used as indices to evaluate the heat decomposition resistance of the materials, as shown in Table 2. The results indicate that the decomposition temperature of PLA did not significantly increase upon the addition of PDLA. This suggests that while the stereocomplexation formed new crystals, the thermal stability was influenced more by the chain structure than by crystalline regions. Consequently, stereocomplexation did not significantly alter the TGA results. Figure 2 presents the TGA curves of thermal decomposition for pure PLA and Sc-PLA/PDLA blends at various compositions. All samples displayed a single-step weight loss, with substantial mass degradation beginning at approximately 303 °C, representing the thermal decomposition of PLA. However, two distinct decomposition steps were observed after PEC was incorporated into Sc-PLA/PDLA blends. The first step, starting at around 303 °C, corresponded to PLA decomposition, while the second step, beginning at approximately 440 °C, was attributed to the decomposition of PEC [14, 22].

Table 2 Summary of comparison of mechanical properties of polymer blends at different composition and sequence mixing.

Sample	T_g (°C)	$T_{m,hc}$ (°C)	$T_{m,sc}$ (°C)	ΔH_m-ΔH_c (J/g °C)	T_{d10} (°C)	T_{d50} (°C)
Neat PLA	62	151	-	12.36	333	358
Sc-PLA/PDLA (95/5)	62	149	207	18.08	335	359
Sc-PLA/PDLA (90/10)	62	148	209	34.34	334	359
Sc-PLA/PDLA (85/5)	62	148	214	44.72	334	359
Sc-PLA/PDLA/ PEC	62	149	208	28.40	334	360
Sc-PLA/PDLA add PEC	62	149	209	28.28	336	361
Sc-PLA/PEC add PDLA	62	149	210	23.51	337	361

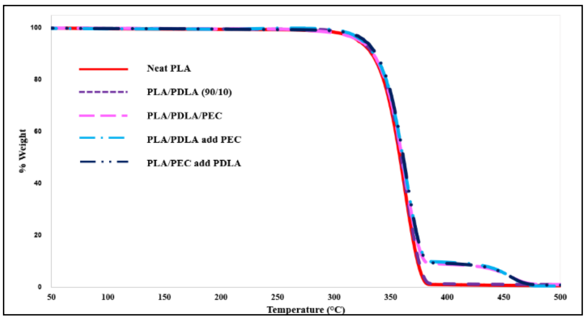

Fig. 2 TGA thermograms of polymer blends at different composition and sequence mixing.

3.4 Crystallinity

The formation of stereocomplex crystallites might be easily detected by X-ray diffraction analysis (XRD). The XRD patterns of Sc-PLA blends as a function of PDLA content and sequences of PEC blends are shown in Fig. 3. Obviously, for the composites without PDLA just, only the width of the amorphous radius could be observed. However, with the incorporation of PDLA 5 wt%, three weak, still, visible aggregation peaks appeared at 2θ values of 11.6º, 20.6º, and 23.5º [2], which were set to the (110), (300)/(030) and (220) planes of stereocomplex crystallites, respectively. Moreover, by increasing PDLA contents up to 15 wt%, the intensity of the three characteristic diffraction peaks became stronger, indicating an enhanced content of stereocomplex crystallite. Moreover, with further adding PEC elastomer in the blends, the intensity of the first diffraction peak at 2θ values of 11.6º became weak. It can be indicated that the PEC elastomer was affected on the (110) plane of the stereocomplex, while it was not involved on the (300)/(030) and (220) planes of the stereocomplex [23]. In addition, the PEC elastomer also decreased the amount of stereocomplex crystallinity in the blends. In addition, XRD could determine the degree of crystallinity in polymers. The degree of crystallinity of neat PLA, Sc-PLA/PDLA, and Sc-PLA/PDLA/elastomer blends are presented in Table 3. The degree of crystallinity was calculated in the following Eq. (1).

$$\% \text{ Crystallinity} = (I_{crystalline} / I_{total}) \times 100 \qquad (1)$$

I_{total} was the area of the total peaks, and $I_{amorphous}$ was the area of the amorphous peak. The degree of crystallinity of neat PLA, Sc-PLA/PDLA (90/10), and Sc-PLA/PDLA/elastomer blends at different sequence mixings are shown in Table 3. The results showed that the degree of crystallinity of PLA increased when adding PDLA and decreased when the Sc-PLA/PDLA blends were added with PEC elastomer. This result indicated that the PDLA might have improved the degree of crystallinity of samples, but PEC elastomer interrupted the crystal formation. However, compared with the sequence mixing of Sc-PLA/PDLA/elastomer blends, the Sc-PLA/PDLA/PEC blends had a similar degree of crystallinity to Sc-PLA/PDLA add PEC but significantly higher than the PLA/PEC add PDLA blends. This result indicated that the sequence mixing of Sc-PLA/PDLA/elastomer blends affected the degree of crystallinity of the polymer.

Table 3 Summarized of the comparison of mechanical properties of polymer blends at different composition and sequence mixing.

Sample	$I_{crystalline}$	I_{total}	$\%X_c$
Neat PLA	0	18424.7	0
Sc-PLA/PDLA (95/5)	252.6	16333.9	1.5
Sc-PLA/PDLA (90/10)	736.9	16665.8	4.2
Sc-PLA/PDLA (85/5)	1199.2	16759.2	6.7
Sc-PLA/PDLA/PEC	498.9	13837.3	3.5
Sc-PLA/PDLA add PEC	445.8	13548.4	3.2
Sc-PLA/PEC add PDLA	358.8	13114.1	2.7

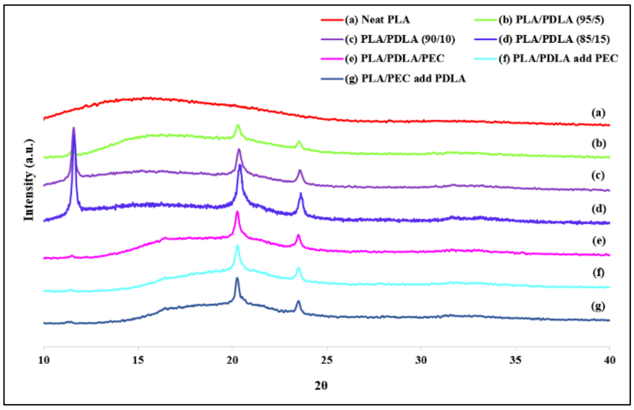

Fig. 3 XRD profiles of polymer blends at different composition and sequence mixing.

4. Conclusion

The purpose of adding elastomer to PLA stereocomplex/elastomer blends was to improve the flexibility of polymer blends. The percent strain at the break meant the polymer's flexibility. The PEC elastomer showed the highest percent strain at the break, which meant it could improve the flexibility of PLA blends as much as possible. Therefore, the PEC elastomer was chosen to be fixed in this study. The content of PEC elastomer was considered in the range of 0 - 15 wt%; at PEC elastomer, 10 wt% could improve the percent strain at break of PLA blends as much as possible. Therefore, the PEC elastomer at 10 wt% was chosen to fix in this study.

Young's modulus, which means the rigid of the polymer, was the main property to consider for choosing the ratio of PLA to PDLA because the Sc-PLA/PDLA blends were mixed with elastomer for study sequence mixing of PLA stereocomplex/elastomer blends. The Sc-PLA/PDLA (90/10) blends had the highest Young's modulus compared to another ratio of PLA to PDLA blends. In addition, the Sc-PLA/PDLA (90/10) blend was the compatibility polymer that SEM confirmed, and it could improve the melting temperature from 148°C of homopolymer to 209°C supported by DSC analysis. Therefore, the ratio of PLA to PDLA blends was chosen for this thesis to be 90 to 10 percent by weight.

The sequence mixing of PLA stereocomplex/elastomer affected the mechanical properties that showed the percent strain at break of Sc-PLA/PDLA add PEC blends was higher than that of Sc-PLA/PDLA/PEC and PLA/PEC add PDLA blends. At the same time, no effect on thermal properties was confirmed by the melting temperature of the stereocomplex, which did not change significantly. However, the sequence mixing affected the degree of crystallinity, and Sc-PLA/PDLA/PEC blends had the highest degree of crystallinity when compared with another sequence mixing.

From the experiment, it can be concluded that the best sequence mixing of PLA stereocomplex/elastomer blends is Sc-PLA/PDLA, which adds PEC because it has the highest mechanical properties. It had the same thermal properties as another sequence mixing, although the degree of crystallinity was unequal. The study of the mixing sequence can be further developed to provide significant benefits to the industrial sector, particularly in reducing production costs and enhancing efficiency. By optimizing the control of the mixing sequence, it is possible to produce high-quality products more effectively.

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