

TOUGHENING OF POLY(LACTIC ACID) BY ETHYLENE-CO-VINYL ACETATE COPOLYMER HAVING VINYL ACETATE CONTENT OF 18%: EFFECT OF ETHYLENE-CO-VINYL ACETATE CONTENT

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Abstract

Poly(lactic acid) (PLA) has received substantial interest recently due to its high strength and stiffness at room temperature. However, it has serious limitations due to brittleness, extremely poor fracture toughness and inferior processability compared to polyolefins. In this research work a possible route to improve the toughness of PLA by blending it with ethylene-co-vinyl acetate copolymer (EVA) having vinyl acetate content of 18%. The compatibility and phase morphology of the PLA/EVA blend system was controlled by the ratio of PLA/EVA. PLA/EVA blends of 100/0, 75/25, 50/50, 25/75 and 0/100 were prepared by extrusion and injection molding; and were characterized for mechanical, thermal, rheological and morphological properties. PLA/EVA blend of 50/50 demonstrated optimized improvement in elongational (28 times) and impact (2 times) properties, attributed to improved compatibility between them due to the sufficient supply of polar vinyl acetate group by EVA to interact with the polymeric structure of PLA.

Keywords

poly(lactic acid), EVA, elongation, morphology, toughness

1. INTRODUCTION

Poly(lactic acid) (PLA), a biodegradable and biocompatible polymer, has received substantial attention in recent years due to its certain favorable properties like high strength and stiffness at room temperature. [1-3] However, it has serious limitation of noteworthy brittleness, extremely poor fracture toughness and inferior processability compared to polyolefins. [4-6] PLA has wide application in packaging, however, the above mentioned problems limits its usage.

Many techniques have been explored to overcome the problem of brittleness, such as plasticization [3-6], copolymerization [7, 8] and blending with other polymers [9-12]. Among them, blending with other polymers was found to be the most effective technique to modify PLA. Various polymers like polyethylene, ethylene-co-vinyl acetate copolymer (EVA), polyurethane, poly(butylene succinate), poly(caprolactone), poly(hydroxyl alkanoates), poly(butylene adipate-co-terephthalate), polyethylene acrylate have been reported to be utilized to improve the performance properties of PLA. However, the obtained improvement in toughness were not

highly substantial.

Afrifah and Matuana improved the toughness of PLA by about 5 times on blending with 30 % (w/w) of ethylene-acrylate copolymer. [13] Ho et al. utilized PLA-polyethylene block copolymer to improve compatibility between PLA and poly(ethylene-co-octene). [14] Semba et al. [12] compatibilized PLA with poly(caprolactone) using dicumyl peroxide, a free radical initiator, to bring about in situ compatibilization; whereas, similar method was utilized by Wang et al. [15] to compatibilize PLA with poly(butylene succinate). Ma et al. [16] investigated the effect of vinyl acetate content to increase compatibility between PLA and EVA. They found that EVA containing 50% vinyl acetate content to be best compatible with PLA. On the contrary, Li et al demonstrated that EVA containing 28% vinyl acetate content to have best compatibility with PLA.

The prime objective of this research paper is to provide simple yet effective method to improve the toughness of PLA by blending it with EVA, having vinyl acetate content of 18% in various weight proportions, using co-rotating twin-screw extruder, having L/D ratio of 32:1. EVA containing vinyl acetate content of 18% is commercially available and thus is cheap. The effect of EVA content on the mechanical, thermal, rheological, crystallinity and morphological properties of PLA/EVA blend is investigated in the current research.

2. MATERIALS AND METHODS

2.1 Materials

PLA (2002D, $M_n = 110$ KDa and $M_w/M_n = 1.7$, l-lactide content of ~96%) was procured from Natureworks LLC. USA. Ethylene-co-vinyl acetate copolymer (EVA1802, vinyl acetate content = 18%, density = 0.93 g/cm^3 , MFI = $2 \text{ g/10 min @ } 190 \text{ }^\circ\text{C/ } 2.16 \text{ Kg load}$) was supplied by Reliance Industries Pvt. Ltd., Mumbai, India. All materials were used as obtained without any modification or purification.

2.2 Preparation of PLA/EVA blend compositions

PLA and EVA were dried in air-circulating oven at $50 \text{ }^\circ\text{C}$ for 10 h, before use. Prepared PLA/EVA compositions are listed in Table 1. All the mentioned compositions were dry blended in a tumbler mixer for 10-15 min and then melt blended in a co-rotating twin screw extruder (APV Baker, UK) having L/D ratio of 32:1, divided into 5 heating zones and a die. Temperature of the extruder zones from hopper to the die was maintained as 90, 110, 130, 150, 170 and $190 \text{ }^\circ\text{C}$ respectively. Co-rotating twin-screw rotation speed and feeder screw rotation speed was maintained constant at 100 rpm and 10 rpm respectively. Obtained extrudates were cooled (chilled water: $10 \text{ }^\circ\text{C}$) and pelletized. Pellets were again dried in air-circulating oven at $50 \text{ }^\circ\text{C}$ for 10 h before further processing. Dried pellets were compression molded to obtain sheets having dimension of $15 \text{ cm} \times 15 \text{ cm} \times 2 \text{ mm}$ for further testings. Compression molding was performed in a compression molding machine having upper and lower mold temperatures as 200 and $210 \text{ }^\circ\text{C}$ respectively, and at a pressure of 15 MPa. Pressure and heat was applied for 20 min during the compression stage, followed by cooling to room temperature which took about 45 min. Obtained sheet (2 sheets per composition) was then cut to get samples for mechanical, thermal, rheological and morphological testing. Cut samples were conditioned for 48 hours before testing at RH of 75% and $23 \text{ }^\circ\text{C}$ temperature.

Table 1. Prepared PLA/EVA compositions

Sr. No.	Nomenclature	PLA		EVA (g)	
		%	g	%	g
1.	PE100/0	100.0	500.0	0.0	0.0
2.	PE75/25	75.0	375.0	25.0	125.0
3.	PE50/50	50.0	250.0	50.0	250.0
4.	PE25/75	25.0	125.0	75.0	375.0
5.	PE0/100	0.0	0.00	100.0	500.0

3. CHARACTERIZATION AND TESTING

3.1 Mechanical properties

Tensile properties (tensile strength, tensile modulus and percentage elongation at break) were determined using a Universal Testing Machine (LR-50K, Lloyds Instrument, UK), according to ASTM standard D638 at a crosshead speed of 50 mm/min. Flexural properties (flexural strength and flexural modulus) were also measured using the Universal Testing Machine at a speed of 1.8 mm/min as per ASTM D790. The notch for impact test was made using a motorized notch-cutting machine (Polytest model 1, Ray Ran, UK); while, the impact strength was determined, as per ASTM D 256, using impact tester (Avery Denison, UK) having striking velocity of 3.46 m/s, employing a 2.7 J striker.

3.2 Thermal properties

Differential Scanning Calorimetric (Q 100 DSC, TA instruments Ltd., India) analysis was undertaken to understand the melting and crystallization behaviour of the prepared blends. Two consecutive heating scans were determined to minimize the influence of possible residual stresses in the material due to any specific thermal history. Scanning rate of 10° C/min was maintained constant for the cycles; whereas, the nitrogen gas purge rate was maintained at 50 ml/min.

3.3 Rheological properties

Melt viscosity (Pa.s) vs shear rate (s^{-1}) and damping factor vs angular frequency (s^{-1}) were measured using a rotational rheometer (MCR101, Anton Paar, Germany) with parallel plate assembly of diameter 35 mm. Samples were pre-dried before analysis. The viscosity was determined against shear rates from $0.01s^{-1}$ to $100s^{-1}$ and damping factor was determined against angular frequencies from $500 s^{-1}$ to $0.5 s^{-1}$ at the temperature of 190°C.

3.4 Morphological properties

Scanning electron microscopy (SEM) analysis was performed with JEOL 6380 LA (Japan). Samples were fractured under liquid nitrogen to avoid any disturbance to the molecular structure and then sputtered with platinum before imaging.

4. RESULTS AND DISCUSSION

4.1 Mechanical properties

Mechanical properties - tensile strength (A), tensile modulus (B), percentage elongation at break (C), flexural strength (D), flexural modulus (E) and impact strength (F) values obtained for the prepared PLA/EVA blend compositions are illustrated in Figure 1. It was found that tensile strength, tensile modulus, flexural strength and flexural modulus decreased, while, elongation at break and impact strength increased with increased concentration of EVA in PLA. However, the values of percentage elongation at break and impact strength of the blend compositions remained nearly constant from PE50/50 composition onwards; however, they were higher compared to PE75/25 and PE100/0. Tensile strength, tensile modulus, flexural strength and flexural modulus decreased drastically for PLA/EVA compositions containing higher proportion of EVA as compared to PE50/50. Thus, this particular composition of PLA and EVA (PE50/50) was found to be optimized composition having best possible improvement in the elongation property without much hampering the other properties.

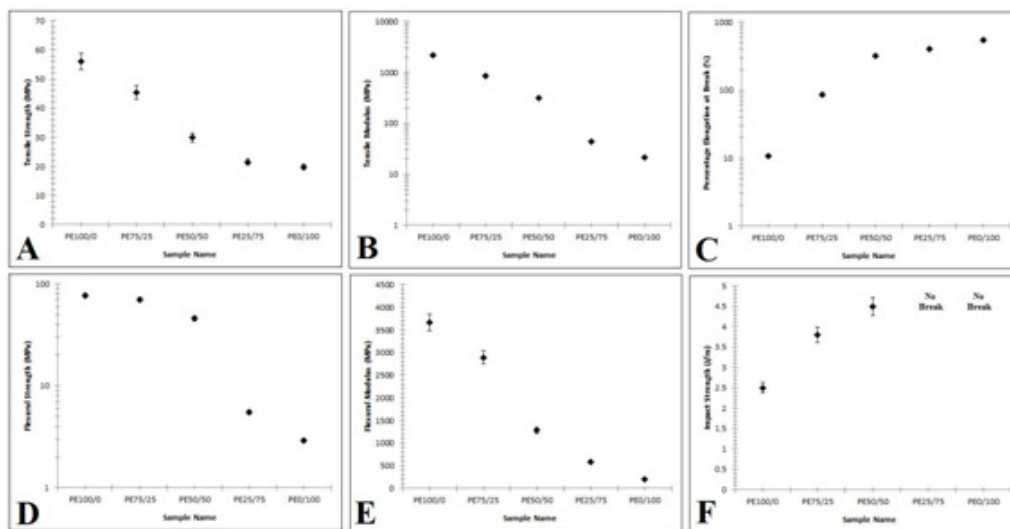


Figure 1. Mechanical properties obtained for the prepared PLA/EVA blend systems

In PE100/0 and PE75/25, higher proportion of PLA suppressed the effect of EVA addition; whereas, in PE25/70 and PE100/0, higher proportion of EVA suppressed the effect of PLA. Thus, PE50/50 was found to be the best composition showing the properties of both EVA and PLA. However, the values of flexural strength and flexural modulus decreased appreciably with increase in concentration of EVA in PLA, which is attributed to the reduction in stiffness supposed to be caused due to the increased flexibility induced by the addition of EVA in PLA. Thus, the reduction in the values of flexural strength and flexural modulus are very good indicators of the increase in flexibility caused by EVA in to PLA.

EVA being elastomeric in nature had very high molecular weight and large number of coiling and molecular entanglements into its molecular structure. This coiled and entangled structure of EVA on mixing with PLA affected the molecular arrangement of PLA polymeric chains, reducing its

molecular packing per unit area. This led to the decrease in the crystallinity and thus the load handling capacity of the blend. Higher the concentration of EVA in PLA, lesser was the load handling capacity of the blend, decreasing the strength and modulus values.

The vinyl acetate content of EVA is polar in nature compared to the ethylene part and is present in 18% proportion. PLA is a known hydrophilic polymeric material. Thus, the only way through which interaction can be brought between PLA and EVA is through the vinyl acetate part of EVA. PE75/25 had lower concentration of EVA, which in turn had lower vinyl acetate content to bring about proper interaction with PLA, demonstrating lower compatibility. Whereas, even though PE25/75 provided higher concentration of EVA, which in turn provided higher vinyl acetate content to interact with PLA, but the EVA structure also induced large number of coiled polymer structure into PLA, making it highly flexible, thus drastically reducing its strength and modulus properties, making it comparable to PE0/100. [17, 18]

4.2 Rheological properties

Figure 2 is a plot of viscosity vs shear rate (A), storage modulus vs angular frequency (B), loss modulus vs angular frequency (C) and damping factor vs angular frequency (D) obtained for the prepared PLA/EVA blend compositions.

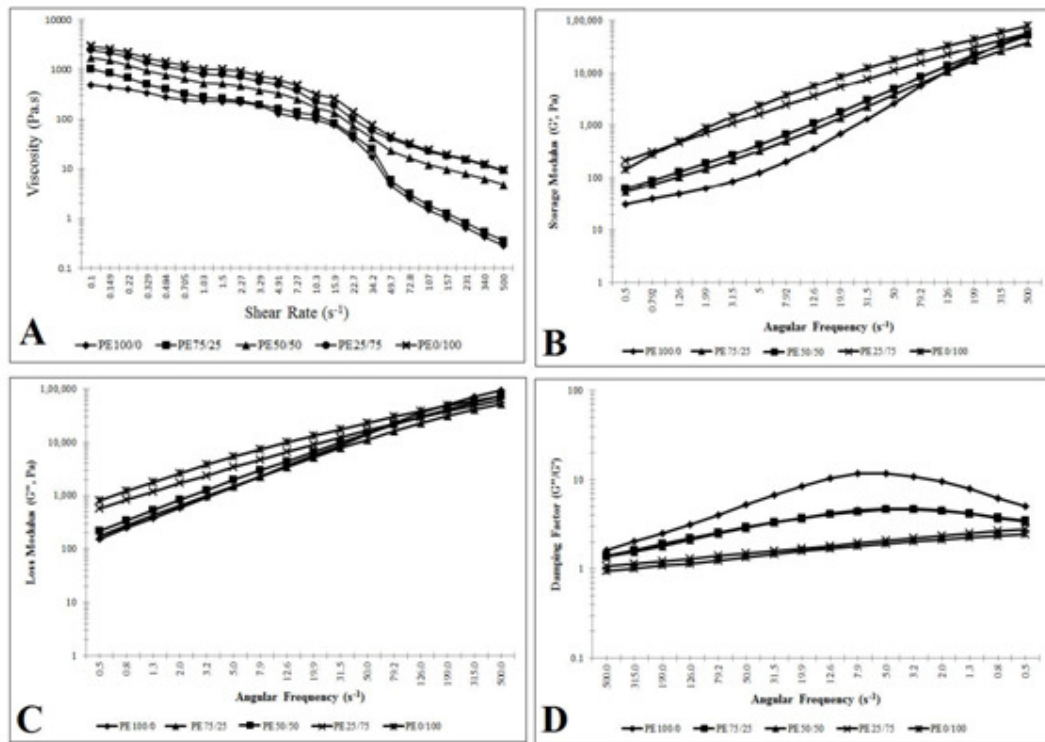


Figure 2. Rheological properties obtained for the prepared PLA/EVA blend systems

From Figure 2 (A) it was determined that the viscosity of all samples decreased with increase in shear rate. Thus, all the samples demonstrated non-Newtonian, shear thinning behavior. [19-22] However, viscosity increased with increased addition of EVA in PLA. Secondly, it was seen that

the viscosity of PE100/0 and PE75/25 decreased appreciably after the shear rate of about 15 s^{-1} , which was not seen in the samples containing higher proportion of EVA. The not so entangled and uncoiled structure of PLA was unable resist the rotational forces exerted by the rotational motion of the rheometer spindle, leading to the rupture of the molecular arrangement at a certain high shear rate, about 15 s^{-1} in our case. This particular shear rate shifted to a slightly higher shear rate on 25% addition of EVA and was completely absent for compositions made with higher concentration of EVA. Peculiar humps were observed in the shear rate range of 1 to 35 s^{-1} . Intensity of this peculiar hump decreased with increased concentration of EVA and is nearly absent in the composition PE0/100. This was supposed to be present due to PLA, demonstrating its drastic viscosity decrease effect as compared to EVA. This demonstrates that processing of EVA is better as compared to PLA due to the non-fluctuating nature of viscosity with shear rate, not requiring close monitoring on the flow behavior of the material.

The coiled and entangled nature of EVA polymeric chains exerted more resistance to the rotating rheometer spindle as compared to not so entangled and coiled structure of PLA polymeric chains. Number of such entangled and coiled polymer chains of EVA increased with increased concentration of it in PLA, increasing viscosity. Secondly, it can be seen that the viscosity curve obtained for PE50/50 is more uniform compared to other curves. Also, the erratic flow nature of the compositions made with higher proportion of PLA is nullified in the PE50/50 composition. Thus, even rheological data corroborates the data obtained from the mechanical properties analysis. Thus, PE50/50 was supposed to be the best PLA/EVA composition demonstrating better elongational as well as rheological performance.

Figure 2 (B and C) demonstrates that storage and loss modulus of PLA/EVA blend increased with increased concentration of EVA in PLA. However, the increase in storage modulus is more appreciable as compared to loss modulus, attributed to the elastomeric nature of EVA. Damping factor is a ratio of loss modulus to storage modulus. It can be seen from Figure 2 (D) that damping factor decreased with increase in concentration of EVA in the PLA/EVA blend system, which is caused due to the more drastic increase in storage modulus values as compared to loss modulus values. From the plots of loss modulus vs angular frequency, storage modulus vs angular frequency and damping factor vs angular frequency, it can be seen that plot of PE50/50 is more linear as compared to the plot obtained for other PLA/EVA compositions. This can be attributed to the improved compatibility between PLA and EVA caused due to sufficient supply of vinyl acetate group by EVA to interact with PLA and bring about best possible compatibility between PLA and EVA.

4.3 Thermal properties

Figures 3 and 4 illustrate the melting and cooling curves obtained for PLA/EVA blend compositions; whereas the values of melting temperature (T_m), enthalpy of melting (H_m), crystallization temperature (T_c) and enthalpy of crystallization (H_c) are listed in Tables 2 and 3, for PLA and EVA, respectively.

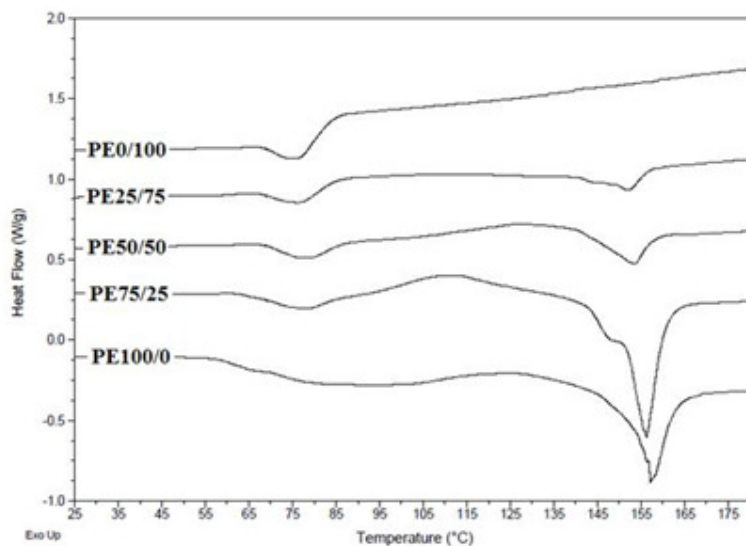


Figure 3. Melting curves obtained for the prepared PLA/EVA blend systems

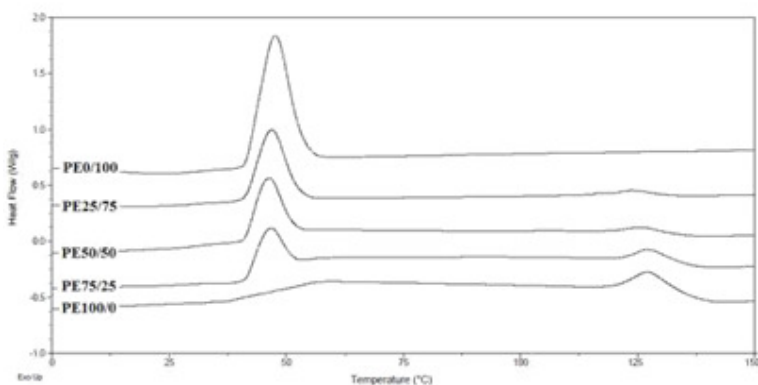


Figure 4. Cooling curves obtained for the prepared PLA/EVA blend systems

Table 2. Thermal properties obtained for PLA in the prepared PLA/EVA blends

Sample Name	T_m (°C)	H_m (J/g)	T_c (°C)	H_c (J/g)
PE100/0	157.5	20.6	128.8	12.7
PE75/25	156.2	19.5	127.3	9.8
PE50/50	154.4	6.2	125.9	7.8
PE25/75	157.5	5.8	125.8	5.7
PE0/100	-	-	-	-

Table 3. Thermal properties obtained for EVA in the prepared PLA/EVA blends

Sample Name	T _m (°C)	H _m (J/g)	T _c (°C)	H _c (J/g)
PE100/0	-	-	-	-
PE75/25	78.1	3.1	46.5	6.9
PE50/50	78.9	3.3	46.3	9.5
PE25/75	77.2	4.1	46.9	12.5
PE0/100	76.5	5.5	46.3	25.1

It was determined that enthalpy of melting (H_m) and enthalpy of crystallization (H_c) of PLA decreased, whereas, that of EVA increased with increase in concentration of EVA in PLA. The shift of melting temperatures of PLA and EVA towards each other suggests some level of interactions happening between the two polymers. Lowest difference between the melting temperatures of the two polymers was obtained for the PE50/50 blend composition containing 50% EVA and 50% PLA. Thus, in correlation with the mechanical properties even through thermal properties it can be interpreted that PE50/50 containing 50% EVA and 50% PLA blend composition is the best PLA/EVA composition demonstrating maximum possible compatibility with each other, caused due to sufficient supply of vinyl acetate group by EVA to interact with PLA.

H_m and H_c of PLA decreased, while that of EVA increased with increase in concentration of EVA in the PLA. This was due to the reduction in the number of PLA molecular chains, requiring lesser amount of energy to melt and thus releasing lesser heat during crystallization, sample quantity remaining near about same. Whereas; the number of molecular chains of EVA increased with increase in its concentration in PLA, requiring higher amount of energy to melt and thus releasing more energy during crystallization.

4.4 Morphological property

Figure 5 illustrates the scanning electron micrographs obtained for PE75/25 (A), PE50/50 (B) and PE25/75 (C) PLA/EVA blends taken at 1000x. Two phases of PLA and EVA can be very easily distinguished from the obtained micrographs. The globular discrete phase demonstrates the rubber phase, which in our case is EVA; while, the continuous phase demonstrates the PLA matrix.

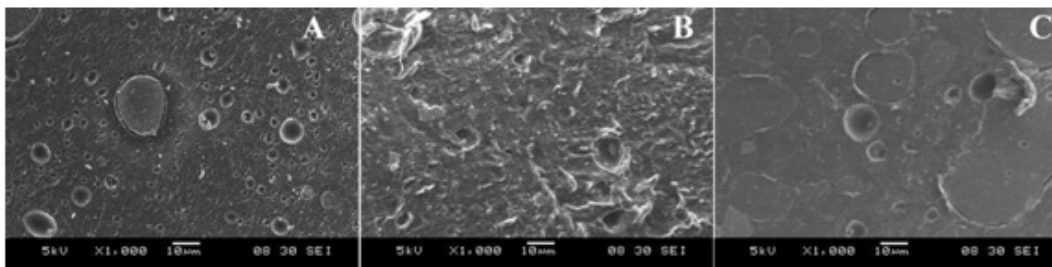


Figure 5. SEM images obtained for (A) PE75/25, (B) PE50/50 and (C) PE25/75

Small and certain big EVA globules can be distinctly seen in (A), while in (C) they can be seen to have formed very big sized globules. (A) consisted of only 25% EVA added in PLA, thus smaller sized and only certain big EVA globules were observed in (A); whereas in (C) EVA is added to about 75% in PLA, making large globules of EVA visible. However, (B) is very interesting to look upon. Globular phase of EVA seems to have got broken down and distributed into the PLA matrix, forming a more uniform matrix. This is caused due to increased compatibility between PLA and EVA due to the sufficient availability of polar vinyl acetate groups in EVA to interact with PLA, showing improved compatibility. Thus, PLA/EVA blend system containing 50/50 proportion of PLA and EVA demonstrated best possible compatibility for EVA containing vinyl acetate content of 18%.

5. CONCLUSION

PLA/EVA blends of various compositions were successfully prepared and characterized; using EVA having vinyl acetate content of 18%, with the motive to reduce the brittleness of PLA and make it easy processable. Prepared blends were characterized for mechanical, thermal, rheological and morphological properties. PE50/50 consisting of 50% EVA and 50% PLA demonstrated best possible compatibility and optimized improvement in the elongational property due to the sufficient availability of vinyl acetate groups by EVA to interact with PLA. Thermal and rheological properties corroborated the optimization obtained in the mechanical properties. Continuous and uniform matrix was observed in PE50/50 due to improved compatibility as compared to two discrete phases been seen in other compositions. Thus, PE50/50 is the best possible composition to be prepared, demonstrating highest possible compatibility, when blending PLA and EVA consisting of vinyl acetate content of 18%.

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