Research Article

Improving the Properties of Biodegradable PLA via Blending with Polyesters for Industrial Applications

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Abstract

Polylactic acid (PLA) is a biodegradable polymer obtained from the fermentation of renewable resources, which can also be used in industrial areas. The employment of PLA is restricted by its brittle nature at room temperature. This study, it is aimed to prepare PLA blends with PBT (Polybutylene terephthalate) to overcome this drawback. Thus, it can be provided new raw materials to many sectors, especially textile, automotive and electronic products, by preparing biodegradable, environmentally friendly materials with better performance properties. PLA/PBT blends containing 5%, 10%, and 15% PBT by weight were produced in a twin screw extruder using with using maleic anhydride grafted ethylene/butyl acrylate (EBA-g-MAH) as a compatibilizer. For comparison studies, blends of PLA/PBT without compatibilizer were also prepared. Examinations on the flow, viscoelastic properties, and rheologic characteristics of prepared samples were carried out by using Melt Flow Index values and Rotational Rheometer measurements. Mechanical and impact resistance characteristics were identified in accordance with the relevant standards. Physical properties of blends such as molding shrinkage, density, and hardness values were also determined. Degradation or cross-linking-induced changes in the chain structure was not observed during the investigation via using a rotational rheometer for PBT samples at 240 °C /15 min., and PLA samples at 260 °C /15 min. Notched izod impact test results show that adding compatibilizer increases 16% impact resistance of PLA/PBT blends. The tensile modulus values of the blends containing compatibilizer decreased by 2% approximately. These results show the transition from brittle to ductile behavior of PLA for compatibilized blends.

Keywords: *Polylactic acid, Polybutylene terephthalate, PLA blends, Compatibilization.*

1. Introduction

Biodegradable polymers have recently attracted significant interest as a replacement for conventional petroleum-based thermoplastics with regard to the effects on the environment because they can be made from a variety of renewable resources and completely decompose via hydrolysis into carbon dioxide and biomass in a short period of time [1,2]. Lactic acid (LA) is polymerized to produce poly(lactic acid) [1,3-5]. PLA is a thermoplastic aliphatic polyester that is produced from non-fossil renewable natural resources by fermentation of polysaccharides or sugar, such as those extracted from corn, potato, cane molasses, sugar beet, etc. This enables the biological cycle to come full circle with PLA biodegradation as well as the photosynthesis process [2-6]. The main uses of PLA have historically been confined to medical applications such as implant devices, tissue scaffolds, internal sutures, and others due to the polymer's high cost, limited availability, and low molecular weight [1-3,4]. High molecular weight PLA has gained a lot of interest as a potential substitute for synthetic polymers in recent years due to new manufacturing techniques that make it possible to produce it affordably [3-7].

Poly(ethylene terephthalate), poly(butylene terephthalate), and poly(trimethylene terephthalate), among other aromatic polyesters, have found extensive use in the packaging and textile industries as well as engineering materials due to their superior thermal and mechanical characteristics [8,9]. Unfortunately, aromatic polyesters have a high level of stability and resistance to deterioration, which is sometimes undesirable, especially for short-term applications [10]. However, biodegradable aliphatic polyesters often have subpar thermal and mechanical properties [8, 11].

Materials containing both aliphatic and aromatic polyesters have received a lot of attention because they have the potential to combine the mechanical performance of aromatic polyesters with the biodegradability associated with aliphatic ones. There are currently some petroleum-based and biodegradable aliphatic-aromatic copolymers with acceptable mechanical properties [11,12]. In light of theoretical calculations, it can be said that PBT and PLA are immiscible. However, at high temperatures, a polyester-polyester mix can undergo three different types of chemical reactions, including alcoholysis, acidolysis, and ester exchange. Block or random copolymers are created as a result of these chemical processes. The catalyst concentration, mixing period, and reaction temperature can all affect the chain structure [13-15].

High molecular weight PLA has gained a lot of interest as a potential substitute for synthetic polymers in recent years due to new manufacturing techniques that make it possible to produce it affordably. The production of partly biobased polymers with improved or desired qualities at a reasonable cost is possible by blending biodegradable polymers with petroleum-based polymers [29,30]. Recently, there has been a lot of interest in the work relating to customizing the characteristics of biobased PLA blends to increase their performances [27-31]. Given that PBT and PLA both belong to the polyester family, they can be evaluated as excellent choices for blending to create a biobased polymer.

Only a limited number of papers have discussed the blends of PLA and PBT. Although the PLA and PBT blend is immiscible, compatibility can be achieved because of the physical interactions between the functional groups of the polyesters [34]. The degradability and crystallization rate of the PLA/PBT mix with para phenylene diisocyanate (PPDI) chain extender were investigated by Kim et al. [35]. They claimed that the PLA phase's crystallization rate had increased and that its degradation rate had slowed down. There are few reports on the transesterification and ring-opening procedures used to polymerize PLA-PBT block copolymers [36]. In recent work, Santos et al. [37] discussed the compatibilizer characteristics of ethylene-glycidyl methacrylate copolymer added to biodegradable PLA in ratios of roughly 3 to 10% by weight.

This study intended to improve the performance characteristics especially the mechanical properties of PLA which is a biodegradable aliphatic polyester. For this objective, melt blending was used to prepare PLA/PBT blends at various compositions, with PLA as the major phase and PBT as the minor phase. Blends of PLA and PBT were compatibilized using an EBA-g-MAH compatibilizer. For comparison investigations, samples of PLA/PBT without any compatibilizer were also manufactured. The rheological properties of neat PLA and PBT were investigated. Mechanical properties of all PLA/PBT blends such as yield strength, elongation at yield, modulus, tensile strength, elongation at break and izod impact strength was determined following the relevant standards. In addition, physical properties such as shrinkage, density, and hardness were also revealed. In light of the obtained results, the effect of PBT addition to PLA polymer and also the use of compatibilizer in the composition on the above-mentioned properties were examined.

2. Materials and Methods

2.1 Materials

Ingeo™ 2003D (NatureWorks), a commercial grade of polylactic acid (PLA) with a melt flow rate of 6 g/10 min. and commercial grade of polybutylene terephthalate (PBT) (TH6095 coded), provided by Xinjiang Blueridge Tunhe Chemical Industry Co. Ltd. with a melt flow rate of 45 g/10 min. were used to prepare PLA/PBT blends. Some selected properties of neat PLA and PBT were given in Figure 1. A chemically modified ethylene butyl acrylate copolymer grafted with maleic anhydride (EBA-g-MAH) Lucofin 1492M HG (LUC) provided by Lucobit (Wesseling, Germany) with a melt flow rate of 5 g/10 min was used as compatibilizer agent. The properties of the compatibilizer agent were given in Figure 1 and the chemical structures of neat PLA, PBT, and compatibilizer agent EBA-g-MAH were shown in Figure 1.

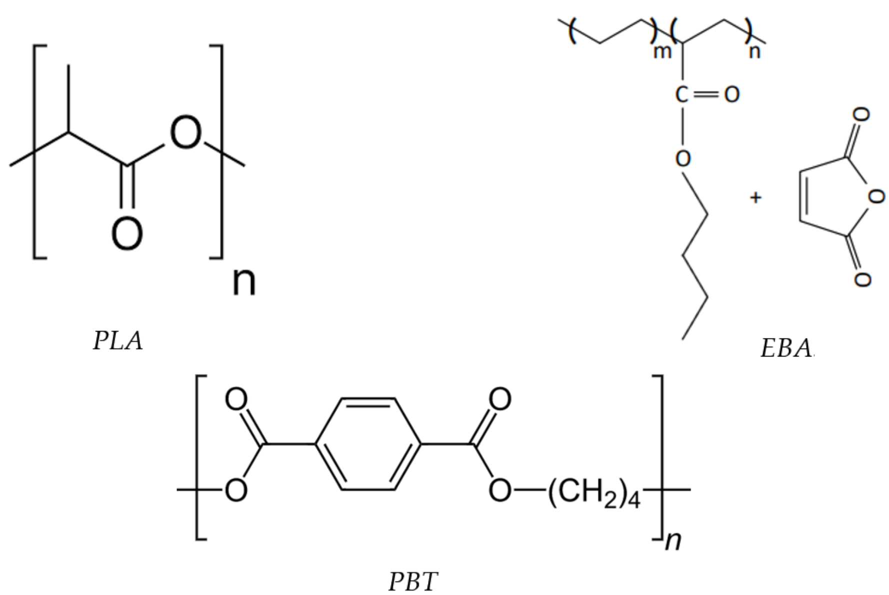

Figure 1. Chemical structures of PLA, PBT and compatibilizer agent EBA-g-MAH.

Table 1. Mechanicals and physical properties of PLA and PBT polymers.

Property	Standard	PLA	PBT	Unit
Density	ISO 1183	1,25	1,3	g/cm³
MFI (190°C /2,16 kg)	ISO 1133	2,66	-	gr/10′
MFI (250°C /2,16 kg)	ISO 1133	-	36	gr/10′
Notched Izod Impact Test	ISO 180/1A	3,81	5,52	kJ/m²
Molding Shrinkage	ISO 294-4	0,42	0,8	%
Tensile Strength	ISO 527-2	75	36	MPa
Elongation at Break	ISO 527-2	2,36	2	%
Yield Strength	ISO 527-2	79,25	58,4	MPa
Elongation at Yield	ISO 527-2	2,21	4	%
Young Modulus	ISO 527-2	3000	2380	MPa
Shore D	ISO 868	73	75	Shore D

Table 2. Properties of EBA-g-MAH compatibilizer.

Typical Analysis	Standard	EBA-G-MAH	Unit
Density	ISO 1183	0,94	g/cm³
MFI (190°C /2,16 kg)	ISO 1183	2,01	gr/10′
Grafting level - Maleic anhydride (bonded)	FTIR	0,76	%

2.2 Rheological Tests

Melt flow index (MFI) values of PLA, PBT, EBA-g-MAH, and PLA/PBT blends were determined by using a Zwick/Roell type BMF-001 plastometer according to ISO 1133 standard. All measurements were performed under the same weight (2.16 kg) but various temperature values proper to the polymer type, 190 °C for both PLA and EBA-g-MAH, 220 °C for PLA/PBT blends, and 250 °C for PBT were chosen. A controlled strain rheometer (AR-G2, TA Instruments) equipped with a 25 mm diameter parallel plate geometry was used for rheology examinations of neat PLA and PBT polymers. Samples were loaded directly between the plates and molded. In the time sweep test, viscoelastic properties of PLA and PBT have been followed at constant strain amplitude (1%) and frequency (1 rad/s) for 15 minutes at 240 °C for PBT and 260 °C for PLA.

2.3 Preparation of PLA/PBT Blends

Melt blending was performed using an intermeshing, corotating twin-screw extruder, having a screw diameter of 30 mm and L/D ratio 36. PLA/PBT blends were prepared with 5%, 10%, and 15% by weight of PBT, 3% by weight of compatibilizer agent, and PLA. Containing no compatibilizer agent samples were also prepared to compare the properties of blends. Extruder zone temperatures between 220-230°C, feed rate 30 kg/h, and screw speed 260 rpm were chosen.

Samples were coded according to component % weight and at the order of PLA/PBT/EBA-g-MAH respectively, for instance, B (92:5:3) coded blend contains 92% PLA, 5% PBT, and 3% EBA-g-MAH. The compositions of PLA/PBT blends were given following in Table 3.

Table 3. The compositions of the PLA/PBT blends.

Samples	PLA	PBT	EBA-g-MAH
B (95:5:0)	95	5	-
B (92:5:3)	92	5	3
B (90:10:0)	90	10	-
B (87:10:3)	87	10	3
B (85:15:0)	85	15	-
B (82:15:3)	82	15	3

2.4 Mechanical Tests

Mechanical tensile tests were performed at room temperature according to ISO 527-2, using a Zwick Roell Z030. The tensile tests of three specimens were conducted at 50.0 mm/min speed and the results were given as average.

Izod impact values were determined by Zwick-Roell HIT 5.5P Impact Tester Pendulum apparatus according to ISO 180/1A standard. Three specimens were tested for all materials at room temperature and 50 % ± 10 relative humidity and the results were given as average.

2.5 Physical Tests

The density values of neat PLA, PBT polymers, and PLA/PBT blends have followed with ISO 1183 standard test method. For measurement of the density of the polymers and blends, HR-250 AZ analytical balance was used. Three samples were used for each material and the mean value of the density has been reported.

The hardness values of three specimens were evaluated using a Shore D hardness tester (Bareiss PRÜFGERTEBA-G-MAHU) according to ISO 868 standard and the mean value has been given.

The molding shrinkage measurement (parallel to the melt flow direction) was realized according to ISO 294-4 standard after injection molding of samples.

3. Results

3.1 Rheological Examination

Examining the results reveals in Table 5 that PLA/PBT blends have lower viscosity characteristics compared to their constituent components. Thus, instead of using PLA alone, PLA/PBT blends can be use with more ease and success for applications requiring low viscosity, such as injection molding and 3D printing.

Regardless of their composition, when PLA/PBT blends are processed MFI value of the second extrudate was increased because of the molecular weights depreciation due to being subjected to repetitive heat treatment.

Due to the improved homogenization for the second extrudate, a reduction in MFI values was seen in all samples, both with and without compatibilizer, as a result of the elevated PBT ratio.

Table 4. MFI values of PLA and PBT.

	PLA	PBT	Unit
MFI (190 °C / 2,16 kg)	2,66	-	gr/10′
MFI (250°C / 2,16 kg)	-	5,52	gr/10′

Table 5. MFI values of PLA/PBT Blends.

	B (85:15:0)-1.	**B (85:15:0)-2**	**B (82:15:3)-1.**	**B (82:15:3)-2.**	**B (90:10:0)-1.**	**B (90:10:0)-2.**	**B (87:10:3)-1.**	**B (87:10:3)-2.**	**B (95:5:0)-1.**	**B (95:5:0)-2**	**B (92:5:3)-1.**	**B (92:5:3)-2.**
MFI (220 °C/ 2,16 kg)	8,44	13,99	7,3	9,94	11,9	8,31	9,76	10,36	13,3	17,02	13,23	14,67

The viscoelastic characteristics of the PLA and PBT samples were monitored by a time sweep test for 15 minutes at a constant elongation (1%) and frequency (1 rad/s). It was tried to determine whether the polymers had undergone structural degradation during the period of investigation. G' stands for elastic or storage modulus, and G" is for viscous or loss modulus in the time sweep test values.

In the time sweep test that has performed for PBT polymer at 240 °C for 15 minutes was observed that PBT did show no considerable change at G' and G" values as seen in Figure 2. Thus PBT polymer was not carried out any structural damage so PBT can be processed under mentioned conditions without any problems.

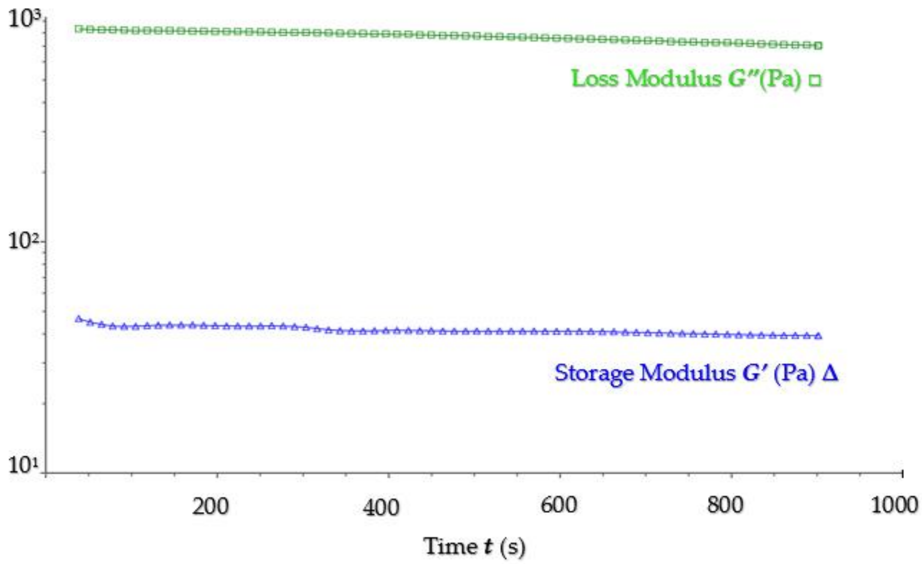

Figure 2. Time sweep test graphic of PBT.

For the time sweep test applied for PLA polymer material, it has been observed a slight change at 240 C for 15 minutes, G' and G" values show a reducing trend as given the following in Figure 3.

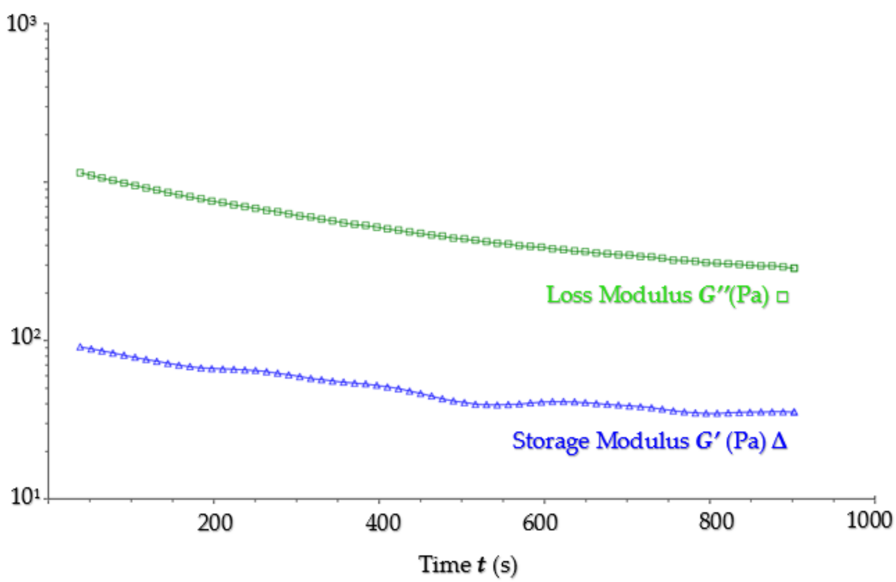

Figure 3. Time sweep test graphic of PLA.

When the related graph is examined it can be seen that decreasing has started in the G' and G" values of PLA was observed after for a period of 200 seconds. Hence it is envisaged that a processing time of 3 minutes for the PLA polymer can be favorable without any destructive effects such as chain degradation, and cross-linking on the structure of the material.

3.2 Mechanical Properties of PLA/PBT Blends

The effect of PBT ratio and compatibilizer agent on the tensile and impact properties in prepared PLA/PBT blends were investigated.

3.2.1 Tensile Properties of PLA/PBT Blends

Considering the tensile properties of PLA/PBT blends containing 15% PBT by weight (Table 6), a noticeable decrease stood out for the blends prepared without compatibilizer, when they were passed through the extruder machine for the second time.

As the B (85:15:0) coded blend which does not contain compatibilizer, for the first extrudate modulus value was 3250 MPa, while the second extrudate decreased to 3140 MPa. Likewise, the tensile strength values of the sample decreased from 64.3 MPa to 56.2 MPa. Tensile strength and elongation at break values were observed also to decrease.

Table 6. Mechanical properties of PLA/PBT blend containing 15% PBT.

Samples	Young Modulus (MPa)	Yield Strength (MPa)	Elongation at Yield (%)	Tensile Strength (MPa)	Elongation at Break (%)
B (85:15:0)-1.*	3250	64,3	2,3	64,3	2,3
B (85:15:0)-2.*	3140	56,2	2	56,2	2
B (82:15:3)-1.	3120	58,9	2,3	58,9	2,3
B (82:15:3)-2.	3130	60,1	2,5	60,1	2,5

* 1. indicates for the first pass and 2. for the second pass extrudate

On the other hand, the tensile properties of B (82:15:3) coded blend containing 3% EBA-g-MAH by weight were improved with second processing via the extruder. Tensile strength values have increased from 58.9 MPa to 60.1 MPa and the elongation at break values has also increased from 2.3% to 2.5%. In summary, by using the compatibilizer, a rising trend was revealed in all mechanical properties with processing via the extruder for the second time.

From these gained results, the importance of using compatibilizers in PLA/PBT blends emerges. For PLA/PBT blends containing 15% PBT by weight, the choice of EBA-g-MAH as the compatibilizer is evaluated as decent. In addition, it can be said that the extruder used in the preparation of PLA/PBT blends cannot effectively mix the PLA and PBT phases in a single-step process, even in the presence of a compatibilizer, but it can be said that blending becomes appropriate with a second processing step.

Table 7. Mechanical properties of PLA/PBT blend containing 10% PBT.

Samples	Young Modulus (MPa)	Yield Strength (MPa)	Elongation at Yield (%)	Tensile Strength (MPa)	Elongation at Break (%)
B (90:10:0)-1.*	3820	67,2	2,5	63,5	3,1
B (90:10:0)-2.*	2990	63,7	2,6	67,3	2,6
B (87:10:3)-1.	3160	59,5	2,3	47,2	5,7
B (87:10:3)-2.	3240	66,5	2,4	66,5	2,4

* 1. indicates for the first pass and 2. for the second pass extrudate

When the properties of PLA/PBT blends containing 10% by weight of PBT were examined (Table 7), a significant decrease was observed in the second extrudate prepared without compatibilizer. It was observed that the first extrudate modulus value of the B (90:10:0) coded blend without compatibilizer decreased from 3820 MPa to 2990 MPa.

Similarly, the tensile values of the sample decreased from 67.2 MPa to 63.7 MPa. Although there is an increase in tensile strength from 63.5 MPa to 67.3 MPa; It is observed that the elongation at break decreased from 3.2% to 2.6% in the same way.

On the other hand, the tensile strength values increased from 59.5 MPa to 66.5 MPa in the second pass through the extruder of the B (87:10:3) coded blend containing 3% EBA-g-MAH. Likewise, an increase was observed in the breaking strength values. The breaking strength value, which was 47.2 MPa, increased to 66.5.

Table 8. Mechanical properties of PLA/PBT blend containing 5% PBT.

Samples	**Young Modulus (MPa)**	**Yield Strength (MPa)**	**Elongation at Yield (%)**	**Tensile Strength (MPa)**	**Elongation at Break (%)**
B (95:5:0)-1.*	3460	69,1	2,3	64,2	2,8
B (95:5:0)-2.*	3360	65,8	2,4	65,8	2,4
B (92:5:3)-1.	3380	62,7	2,2	46,5	6,1
B (92:5:3)-2.	3440	68,3	2,3	53,9	6,4

* 1. indicates for the first pass and 2. for the second pass extrudate

Finally, when PLA/PBT blends containing 5% by weight PBT were examined (Table 8), it was observed that the B (95:5:0) blend without compatibilizer lost its mechanical values in the first and second passes through the extruder.

It is seen that the elastic modulus has decreased from 3460 MPa to 3360 MPa. The tensile strength from 69.1 MPa decreased to 65.8 MPa. However, an improvement was observed in the mechanical values of the prepared blend when it was passed through the extruder a second time. Among other mechanical properties, the tensile strength increased from 62.7 MPa to 68.3 MPa. In the same way, the increase in mechanical properties is; The tensile strength increased from 46.5 MPa to 53.9 MPa.

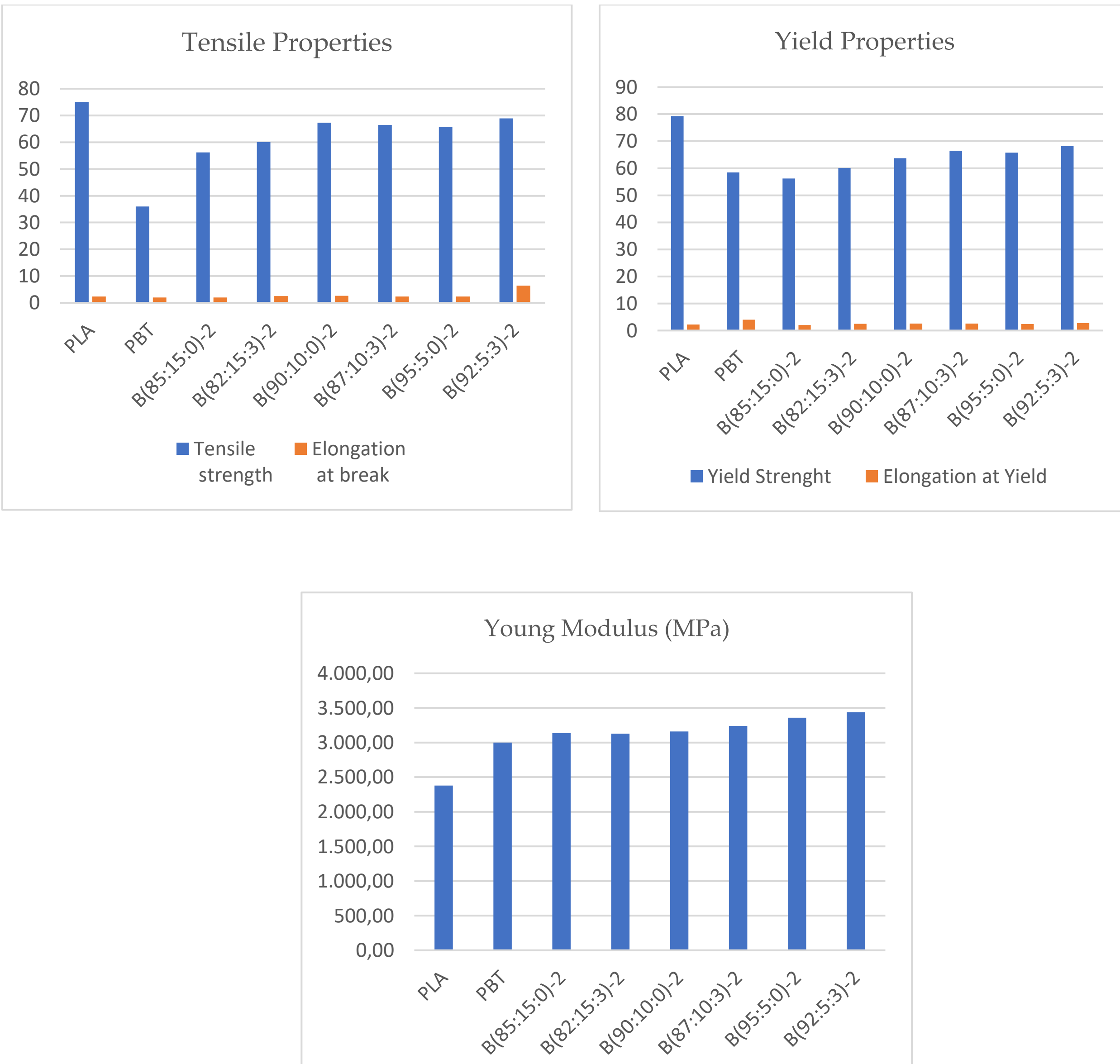

Figure 4. Mechanical test results of PLA, PBT, and PLA/PBT blends.

3.2.2 Notched Izod Impact Test

When we analyze the notched izod impact test analyses in terms of mechanical tests from Table 9, it has been observed that passing through the extruder twice, whether or not compatibilizer is used in PLA/PBT blends, increases the notched izod impact strength values.

Table 9. Impact strenght properties.

	PLA	PBT	B (85:15:0)-1.	B (85:15:0)-2	B (82:15:3)-1.	B (82:15:3)-2.	B (90:10:0)-1.	B (90:10:0)-2.	B (87:10:3)-1.	B (87:10:3)-2.	B (95:5:0)-1.	B (95:5:0)-2	B (92:5:3)-1.	B (92:5:3)-2.
Notched Izod Impact Test (kJ/m²)	3,98	5,52	2,21	2,2	2,54	2,63	2,55	3,15	2,66	3,24	2,57	2,93	3,23	3,51

Especially when the B (92:5:3) blend prepared using 3% EBA-g-MAH, a compatibilizer containing 5% PBT, first passed through the extruder, the notched izod impact value was 3.23 kJ/m²; It was observed that it increased to 3.51 kJ/m² when it was passed through the extruder for the second time.

It was observed that the impact strength values were lower with increasing PBT amount. The best-observed impact properties were obtained with 5% PBT using a compatibilizer.

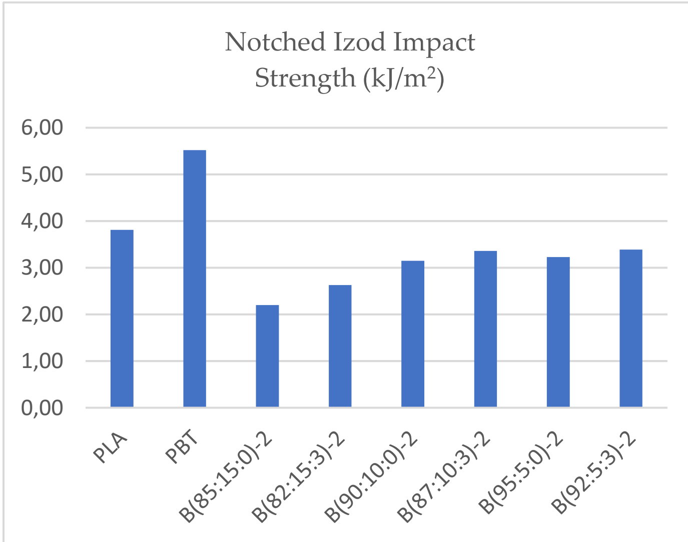

Figure 5. Notched izod impact properties of PLA, PBT, and PLA/PBT blends.

3.3 Physical Properties of PLA/PBT Blends

The effect of the PBT ratio on the physical properties of the PLA/PBT blend in the prepared PLA/PBT blends were investigated. The features examined here are; Tests such as density, hardness, and mold shrinkage were examined and the results are shown in Table 10.

Table 10. Physical properties of PLA, PBT polymers, and PLA/PBT blends.

	PLA	PBT	B (85:15:0)-1.	B (85:15:0)-2	B (82:15:3)-1.	B (82:15:3)-2.	B (90:10:0)-1.	B (90:10:0)-2.	B (87:10:3)-1.	B (87:10:3)-2.	B (95:5:0)-1.	B (95:5:0)-2	B (92:5:3)-1.	B (92:5:3)-2.
Density (g/cm³)	1,3	1,25	1,27	1,27	1,25	1,25	1,26	1,26	1,25	1,26	1,26	1,26	1,25	1,25
Shore D (ShD)	73	75	76	76	74	74	75	75	76	75	75	75	76	76
Molding Shrinkage (%)	0,42	0,8	0,81	0,72	0,64	0,51	0,41	0,4	0,34	0,51	0,41	0,4	0,34	0,45

3.4 Effect of EBA-g-MAH Compatibilizer on the mechanical properties of PLA/PBT Blends

The effects of using EBA-g-MAH as a compatibilizer in PLA/PBT blends have a very important place on impact resistance and tensile properties.

The presence of a compatibilizer is very necessary to increase the impact strength. For example; B (90:10:0)-1. with B (95:5:0)-1. When compared, the izod impact strength increased from 2.55 kJ/m^2 to 2.93 kJ/m^2. However, when a 3% EBA-g-MAH compatibilizer agent was added to these blends, the izod values increased from 2.66 kJ/m^2 to 3.23 kJ/m^2. This may indicate that good interfacial adhesion is required. So the fields can dissipate impact energy when the matrix fails due to fracture. It was observed that the impact strength increased with the increase in the amount of dispersed phase.

In the B (85:15:0) blend prepared without compatibilizer with PLA/PBT blend containing 15% PBT, 3% EBA-g-MAH had no effect on the elongation at break (Table 6).

On the other hand, significant improvements have been noticed in PLA/PBT blends containing 10% and 5% PBT. For example; In the B (90:10:0) blend, the elongation at break was 3.1%; When 3% EBA-g-MAH was used, the elongation at break increased to 5.7% (Table 7).

The addition of compatibilizer had a significant effect on samples B (95:5:0) and B (92:5:3), increasing their elongation at break from 2.8% to 6.8% on their first pass through the extruder (Table 8).

In blends containing 5% by weight PBT, the ethylene concentration is higher than the concentration of the dispersed phase, which makes a high contribution to the increase in elongation at break values.

It was observed that the elongation at break decreased when the ratio of PBT increased. This situation shows that PBT exhibits a fragile behavior when used more than 10%.

4. Conclusion

EBA-g-MAH compatibilizer was used to improve the properties of sustainable biobased polymer blends made from PLA and PBT. When using 3% EBA-g-MAH, PLA/PBT blends were able to outperform the without compatibilizer PLA/PBT blend in terms of notched izod impact strength. The tensile strength, tensile modulus and elongation at break of the blend were improved with the addition of the EBA-g-MAH compatibilizer.

The best samples were determined to be suitable at 5% in terms of strength values among the 15%, 10%, and 5% samples in PLA and PBT combinations. These blends were subjected to heat treatment and decomposed. For this reason, its durability has been increased by adding 3% EBA-g-MAH as a compatibilizer in PLA/PBT blends. In other words, the purpose of these studies were to investigate its use in terms of sustainability and reusability. Additionally, it has been noted that 5% PBT containing PLA/PBT blends have improved in terms of mechanical and notched izod impact strengths. Results of the notched izod impact test show that the addition of compatibilizer increases the impact resistance of PLA/PBT blends by 16%. The significant increase in notched izod impact strength of the blends can be attributed to the EBA-g-MAH compatibilizers interfacial compatibility. The EBA-g-MAH compatibilizers interfacial compatibility is what accounts for the blends notable rise in notched izod impact strength. Tensile modulus values of PLA/PBT blends with 3% EBA-g-MAH compatibilizer were decreased by 2% approximately. According to these investigates, PLA for compatibilized blends changes from brittle to ductile behavior.

In this research, blending polymers is also intended to provide high performance polymer materials for novel uses. By sustainability materials with biodegradable, sustainable, and improved performance features and with it is intended to generate alternative raw materials for several industries, including automotive, electrical, and electronic devices. In addition to all this, it aims to minimize dependence on petroleum-based resources and promote more sustainable development by creating a high toughness and sustainable bio-based PLA/PBT combination.

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