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RESEARCH ARTICLE

Compatibilization of Poly (lactic acid)/Polycarbonate Blends using Triacetin-mediated Interchange Reactions

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Abstract There is a strong interest in the use of poly (lactic acid) PLA which is a renewable polymer for environmental sustainability. The aim of the paper is to improve the properties of PLA by blending with polycarbonate (PC) at various ratios using melt blending technique. The effects of two types of triacetin (TA) as compatibilizers and tetra butyl ammonium tetraphenylborate (TBATPB) as catalyst were investigated. Mechanical, thermal and morphological properties of the PLA/PC blends were examined. The addition of both triacetin (2022 type, 5 wt%) with catalyst (0.2 wt%) in 50PLA/50PC blends showed good improvement in modulus and elongation at break. The Young's modulus, flexural modulus and elongation at break increased by 280MPa, 238MPa and 94% respectively compared to uncompatibilzed 50PLA/50PC blend. It was depicted that transesterification reaction can take place only in the presence of compatibilizers; however, the addition of the catalyst accelerates the reaction. Dynamic mechanical analysis revealed a new peak attributed to the glass transition temperature (T_g) of the PLA-PC copolymer at a temperature (~ 110 °C) higher than T_g of typical PLA and lower than the T_g of typical PC. It can be concluded that the addition of triacetin and catalyst results in the overall best mechanical properties, thereby confirming the synergistic action promoted the transesterification reaction between PLA and PC promoting the compatibility of the PLA/PC system.

Keywords: Poly (lactic acid), polycarbonate, triacetin, catalyst, mechanical analysis, morphological analysis.

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Introduction

There is a strong interest in the use of renewable resources for environmental sustainability. In this context, PLA is a versatile polymer that consists of several unique properties such as biocompatibility, biodegradability, renewable, and eco-friendly nature. It also possesses some alluring properties like high tensile strength, elastic modulus, transparency, stiffness, and resistance to oils [1–2]. Nevertheless, PLA has some drawbacks that are associated with its low thermal stability, low impact & elongation at break, crystallization rate, conductivity, and high ductility, rapid decomposition, hydrolytic susceptibility, and production cost [3]. PC is a group of aromatic polyester consisting of carbonate groups with high level of aromaticity. PC is a strong and tough material due to the presence of the ether linkages in its backbone chain that allows some flexibility and rotation in the polymeric chains [4, 21, 22]. It is a well-known engineering thermoplastic with exceptional properties such as high mechanical strength (E_b% break &

Impact), high thermal stability (T_g, T_m, T_c) and melt viscosity along with polarity [5-6].

The drawbacks associated with PLA can be overcome by blending PLA with PC, which offers the excellent combined properties of the two polymers and prevailing over the individual shortcomings. For example, the thermal stability and the toughness of the PLA can be enhanced by combining with PC. Moreover, the modulus, stiffness and chemical resistance of PC can be increased with the addition of PLA [4]. Hence, nowadays, researchers are more interested in the preparation of such blends and their several end-users applications such as in laptops, cellophanes, and auto parts owing to their biodegradability and excellent mechanical properties [5–7, 20, 21].

However, PC and PLA are immiscible because of their inherent chemical structure, polarity and high melt viscosity. These cause difficulties for the interpenetration of molecular chains of both polymers and resulting in a brittle blends. Therefore, many studies [8–16] were carried out to enhance the compatibility of PLA/PC blend which is a key for various end-user applications. In comparison to synthesising a new copolymer to achieve the desired properties that cannot be achieved with the currently available polymers, melting PC and PLA will be a much more practical and cost-effective technology. The usage of appropriate compatibilizer like triacetin is essential along with the catalyst to improve toughness by enhancing adhesion at the interface of PLA/PC blend [3]. The interfacial strength acts as a significant role in the performances of the polymer blends, and the enhanced strength can be stabilized for the stress transmission, which is found by the degree of interfacial polymer chains entanglement [17–20].

In the present study, an attempt was made to fabricate PLA/PC blends using melt blending technique followed by injection molding. A comparison on the effectiveness of two types of triacetin (TA) as compatibilizers for the blend was done. Besides triacetin, tetra butyl ammonium tetra phenylborate (TBATPB) was used as a catalyst in this research. To the best of our knowledge, no research work has reported on comparing the effectiveness of triacetin 2022 and triacetin 1578. This research is important to further enhance the properties of PLA/PC blends for its usage in different end-user applications. The mechanical tests, thermal properties and surface morphology of the PLA/PC blends were examined to investigate the utility of bio-based blends for different end user applications.

Materials and Methods

Materials

Injection moulding grade, PLA (Nature Work 3052D) with a density of 1.24 g/cc, melt flow index (MFI) of 15g/10min (210 °C, 2.16kg), and glass transition temperature of 50 – 55 °C with average molecular weight of Mw130000 g/mol was purchased from Nature Work LLC® (Minnetonaka US-NM). PC (Panlite® L-1225Y), was obtained from Teijin Chemicals with MFI of 11g/10min, (300 °C, 1.2kg), density of 1.20g/cc and glass transition temperature of 150 – 155 °C. Two types of triacetin were used as compatabilizers; Palmester 2022 (density of 1.154 gm/cc and melting temperature of 171°C) and Palmester 1578 (density of 1.159 gm/cc and melting temperature of 173 °C). They were purchased from Nature Taiko Palm-Oleo® China. Tetrabutylammonium tetraphenylborate (TBATPB) was purchased from Sigma-Aldrich.

Fabrication of Blends

Firstly, the raw materials: PLA and PC were dried at 55 °C for 24-hrs and thereafter mechanically mixed at room temperature in a tumbler for around 10 min in the pre-decided ratios as shown in Table 1 The mechanical mixed polymers were then melt blended using a co-rotating twin screw extruder; Plastics Corder PL2000. For all experiments, the extruder hopper to die temperature range was 200 °C to 230 °C and the screw speed was remained at 65 rpm. After optimization of the PLA/PC blend ratio, the same procedure was followed but with the inclusion of 5% compatibilizer and 0.2% catalyst according to the literature in Figure (1), to enhance the polymer matrix interfacial adhesion. After pelletization, the sample pellets were dried at 80 °C for 24 hrs. Furthermore, the injection molding was carried out by an injection molding machine (JSW 100 Tons, Japan) to produce the sample specimens. The temperature setting from hopper to nozzle was set between 200 °C – 230 °C. The process was carried out at an injection pressure and speed of 40 bars and 35 rpm.

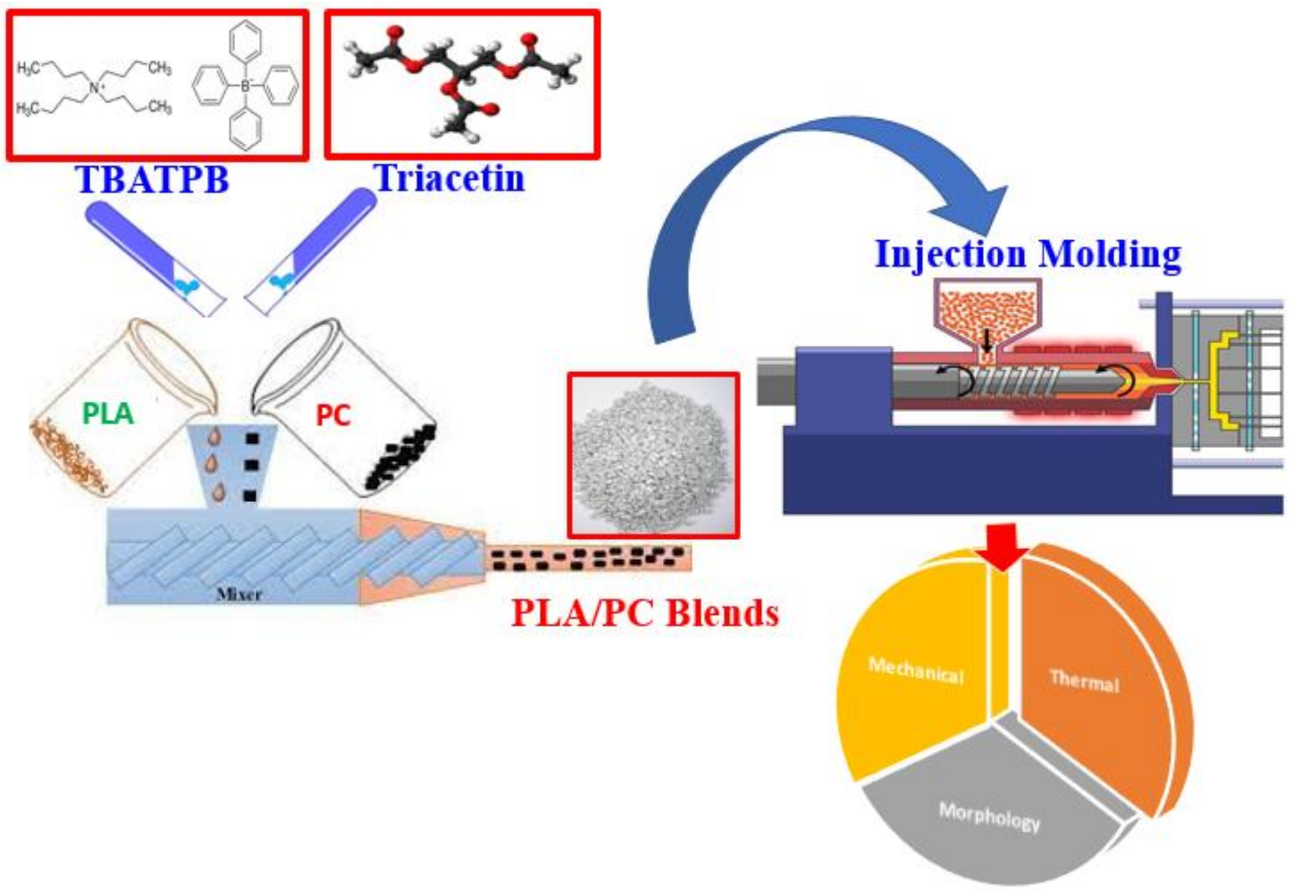


Figure 1. Schematic diagram of the fabrication of blends

Table 1. Composition of PLA, PC, Triacetin and Catalyst.

Sample Name	PLA wt%	PC wt%	CA wt%	TA-2022 wt%	TA-1578 Wt%
PLA	100	-	-	-	-
90PLA/10PC	90	10	-	-	-
80PLA/20PC	80	20	-	-	-
70PLA/30PC	70	30	-	-	-
60PLA/40PC	60	40	-	-	-
50PLA /50PC	50	50	-	-	-
40PLA/60PC	40	60	-	-	-
PC	-	100	-	-	-
50PLA/50PC/0.2CA	50	50	0.2	-	-
50PLA/50PC/5TA-2022	50	50	-	5	-
50PLA/50PC/5TA-1578	50	50	-	-	5
50PLA/50PC/TA-2022/0.2CA	50	50	0.2	5	-
50PLA/50PC/TA-1578/0.2CA	50	50	0.2	-	5

Testing and Characterization

Mechanical Testing

Tensile Testing

Tensile testing was performed using Tensile Tester LLOYD with a load cell of 20 kN in accordance with ASTM D638. The test was performed at room temperature with a fixed crosshead speed of 5mm/min and 100mm gauge length. Five samples were tested for each composition to determine the average values.

Flexural Testing

Flexural testing was performed using Tensile Tester LLOYD with a load cell of 20 kN in accordance with ASTM D790. The strain rate used was 5 mm/min with 100 mm span distance. Five samples were tested for each composition to determine the average values. The test was performed at room temperature.

Impact Testing

Impact testing was performed using GOTECH Impact Tester in accordance with ASTM D256. Five samples were tested for each composition to determine the average values. The test was performed at room temperature.

Thermal Analysis

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was performed using Parkin Elmer YGA7. Approximately 8-10 mg of sample was placed inside the sample holder and scanned at the rate of 10 °C/min under N_2-gas flow from ambient temperature to 600 °C. The thermograms recorded the percentage of weight loss versus temperature.

Dynamic Mechanical Analysis (DMA)

Dynamic Mechanical Analysis (DMA) was used to study the viscoelastic behaviour of polymeric materials at distinct temperatures and frequencies. The analysis was performed using DMA-Q800, M/s TA Instruments, USA, at a frequency of 1 Hz. The temperature used was between -50 °C to 150 °C at a heating rate of 10 °C/min.

Morphological Analysis

Field Emission Scanning Electron Microscopy (FESEM)

Morphological analysis was conducted with Carl Zeiss (Germany) Supra 35 VP microscope with a voltage in the range of 8-10 kV. The impact fractured samples were placed into specimens' stubs and coated with gold by sputtering (Balzers union MED-010-coater) to avoid the build-up of electrostatic charges on the sample surface. The coated samples were observed under FESEM, the micrograph of the fractured surface of neat PLA and PC, and their respective PLA/PC blends.

Transmission Electron Microscopy (TEM)

The TEM micrographs were collected using samples of sizes 0.2 cm X 1 cm which were cut into trapezium shapes on a ZEISS LIBRA-120 Transmission Electron Microscopy, equipped with a Gatan-Ultrascan 1000 2k X 2k CCD-camera operating at an accelerating voltage of 120 kV.

Results and Discussion

Mechanical Properties

Table 2 summarizes the tensile properties of PLA/PC blends as a function of their varied composition in the presence of compatibilizers and catalyst. For instance, the tensile strength and modulus of PLA obtained were 65.02 MPa and 3580 MPa, respectively. Additionally, the elongation at break (%) was found to be 3.50%. Incorporation of PC in the PLA/PC blends did not show any measurable changes in the tensile strength, as the values of tensile strength for PLA and PC are quite similar. The blend prepared at (50/50) wt. % of PLA and PC showed optimum mechanical performance of about 64.88 MPa and 3020 MPa, respectively. The elongation at break (%) of about 6.08% which is about 2.58% higher than PLA was also reported [1-3]. Phuong *et al*. [2] reported similar behaviour of PLA/PC blends where the authors observed no noticeable change in the tensile strength of PLA/PC blends when compared to their respective virgin polymers.

As observed in Table 2, there is no noticeable increment in the tensile properties of PLA/PC blend when compared to their virgin ones. This could possibly be due to the poor adhesion at the interface of PLA and PC, which in turn may create hindrance and differences between the molecular structures and melt viscosity of PLA and PC [22–24]. Based on the test result for the mechanical properties, the blend ratios of (50/50) weight % of 50PLA/50PC is the optimized ratio for the blend optimization and hence, this ratio is considered for the compatibilization with a catalyst and different (TA-2022 & TA-1578) types of triacetin to discover the effects on the properties. Table 2 displays the findings of the mechanical properties measurements using catalyst, triacetin, and both. The interfacial adhesion between the PLA and PC blends was increased by the addition of two types of compatibilizers as stated above in the optimized 50PLA/50PC blend. A comparative study between both the compatibilizers was carried out to optimize the increased adhesion at the interface of the blends. 5wt% of both the compatibilizers was considered in the 50PLA/50PC blends as reported in the literature [25].

50PLA/50PC/5TA-2022 blend depicted an optimum mechanical performance having tensile strength, modulus and elongation at break (%) of about 63.79 MPa, 3380 MPa and 81.55% respectively. It depicts an increase of 1.86%, 3.36% and 204.9% in their respective tensile strength, modulus and elongation % as compared to 50PLA/50PC/5TA-1578 blends. The increase in the tensile properties is majorly due to the improved interface of the PLA/PC blends in the presence of TA-2022 compatibilizers. According to Melia *et al*. [18], the addition of TA-2022 caused a transesterification reaction in the blends, which improved the mechanical properties by increasing compatibility.

The addition of 0.2% CA-TBAPAB catalyst in 50PLA/50PC blend has led to modest increase in the tensile properties of the blends such as tensile strength; 63.05 MPa, tensile modulus; 3290 MPa, and elongation at break; 30.71%, respectively. The addition of both catalyst and TA-2022 in 50PL/50PC/0.2CA/5TA-2022 represented optimum mechanical performance of tensile strength, modulus and elongation at break of 64.92 MPa (an increase of 3.2% with respect to the mechanical mixture), 3300 MPa and 102.34%, respectively. These indicate that both CA-TBAPAB & TA-2022 are chemically active in this system, yielding the best mechanical performance shown by the 50PL/50PC/0.2CA/5TA-2022 blends. The synergistic action of CA & TA in promoting the compatibility of the PLA/PC system is thus confirmed. Similar behaviour is corroborated by morphological analysis where the addition of both TA-2022 and catalyst in 50PL/50PC/0.2CA/5TA-2022 represented the surface morphology without any voids and pits (Figure 2(e)). The improvement in elongation at break signifies that TA-2022 is more effective than TA-1578 triacetin [4–7, 25].

Table 2. Mechanical properties of PLA, PC and its blends with different types

Sample Name	Tensile Strength (MPa	Tensile Modulus (MPa	Elongation @break (%)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Izod Impact Notched (kj/m)
PLA	65.05±2.20	3580±34.52	3.50±0.31	112.40±2.12	3600.±60	3.00±0.02
90PLA/10PC	56.63±1.80	3510±31.20	2.42±0.25	102.64±2.02	3461.±39	2.67±0.017
80PLA/20PC	55.37±2.30	3420±28.52	2.48±0.29	92.13±1.65	3486.±43	2.75±0.017
70PLA/30PC	54.83±1.20	3200±27.77	5.27±0.37	83.01±1.47	3508.±47	2.78±0.017
60PLA/40PC	57.33±1.10	3060±26.35	5.44±0.49	98.85±1.72	3520.±50	3.15±0.021
50PLA /50PC	64.88±1.70	3020±26.12	6.08±1.52	104.32±2.03	3559±58	5.25±0.023
40PLA/60PC	62.07±2.1	2980±25.57	10.04±.72	101.58±1.9	3537±56	6.00±0.033
PC	61.50±1.60	2400±21.89	80.00±2.7	101.58±1.9	2350.±33	71.00±1.1
50PLA/50PC/0.2CA	63.05±1.46	3290±27.05	30.71±1.17	106.56±2.32	3560±31.72	10.25±0.3
50PLA/50PC/5TA-2022	63.79±1.55	3380±28.35	81.55±2.8	107.98±3.11	3579±31.56	3.64±0.02
50PLA/50PC/5TA-1578	62.62±1.33	3270±26.73	26.74±0.95	105.88±2.85	3563±31.02	3.16±0.02
50PLA/50PC/TA-2022/0.2CA	64.92±1.68	3300±27.65	102.34±3.1	111.87±3.49	3598±32.02	3.75±0.02
50PLA/50PC/TA-1578/0.2CA	63.04±1.55	3210±26.01	81.55±2.8	107.98±3.11	3579±31.56	3.64±0.02

Morphological studies

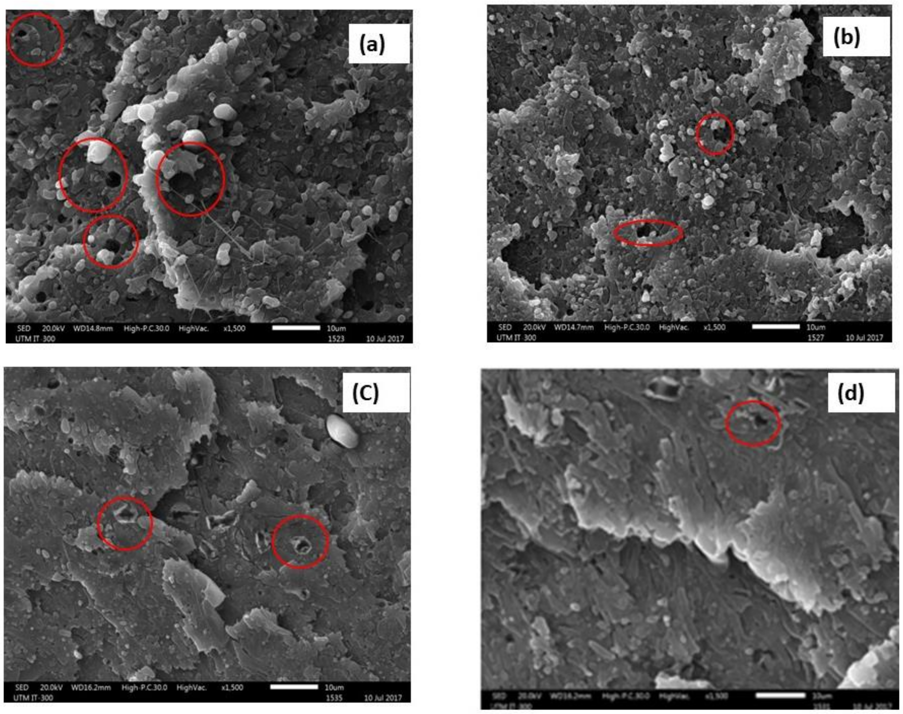
The application of FESEM and TEM in analysing the surface topography has been largely used in characterizing the extent of compatibility and interfacial adhesion of the blends. FESEM micrographs of PLA/PC, and PLA/PC/CA/TA blends were used to study the extent of adhesion of the different phases of the blends. It is expected that the micrographs will expose the influence of triacetin as a compatibilizer in the blends. The addition of triacetin and catalyst is expected to provide good interfacial adhesion in the PLA/PC interface.

FESEM Analysis

FESEM micrographs of the impact fracture surface of PLA/PC blends with and without CA & TA are illustrated in Figures 2 & 3 respectively. In Figure 2 (a) and Figure 3, PLA/PC blends indicate that the adhesion between PLA/PC will be poor as evidenced by the large number of oval cavities in impactfractures samples and behaved, as expected, as immiscible polymer blends. The phase separation is obvious that the PC matrix in the PLA domains can be easily detected. It is noticeable that the presence of large spaces between the PC and PLA domains are attributed to low adhesion which was confirmed by Figure 3(c). The size of the PC domains was variable, in the range of 4-13µm [14–16].

Figure 2(b) represents the morphology of blends in the presence of a catalyst. It is clearly visible that the incorporation of catalyst indicates that the number of voids and pits was decreased as compared to uncompatiblized PLA/PC blends (indicated by red circles). Hence, it indicates that the catalyst is chemically active in this system and the same has been corroborated in the mechanical performance in Table 2. Micrographs in Figures 2(c) and (d) show the surface morphology of the PLA/PC blends in presence of TA-2022 and TA-1578. It is clearly evident that the number of voids and pits is lesser when compared to uncompatiblized blends. Additionally, the size of PC droplets was measured to depict smaller dimensions in the range of 2-10 um. This dispersion may be due to the transesterification mechanism, thereby stabilizing the PC droplets from coalescing [18].

Furthermore, the addition of both TA-2022 and catalyst in 50PLA/50PC/0.2CA/5TA-2022 represented the surface morphology without any voids and pits as indicated by the yellow circles (Figure 2(e)). These indicate that both CA-TBAPAB and TA-2022 are chemically active in this system, and similar behaviour is being depicted through mechanical performance as shown by the 50PLA/50PC/0.2CA/5TA-1578 blends (Figure 2(f)). Therefore, the synergism of CA & TA in promoting the compatibility of the PLA/PC system is confirmed [26].



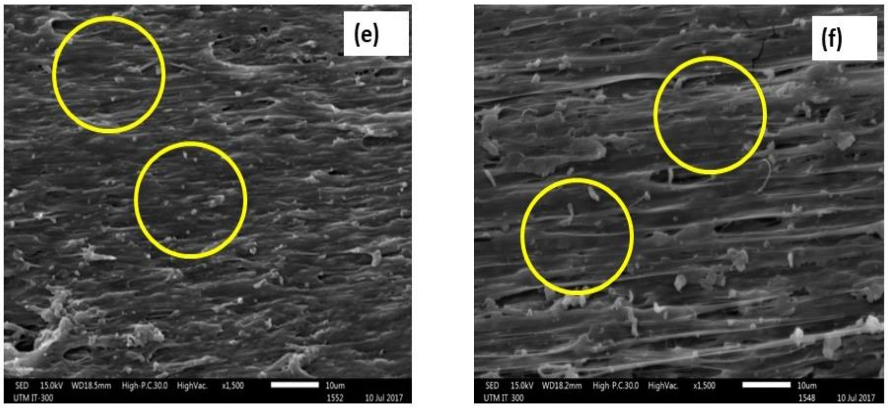


Figure 2. FESEM micrographs of impact-fractured surface (a) 50PLA/50PC (b) 50PLA/50PC/0.2CA (c) 50PLA/50PC/5TA-2022 (d) 50PLA/50PC/5TA-1578 (e) 50PLA/50PC/0.2CA/5TA-2022 (f) 50PLA/50PC/0.2CA/5TA-1578

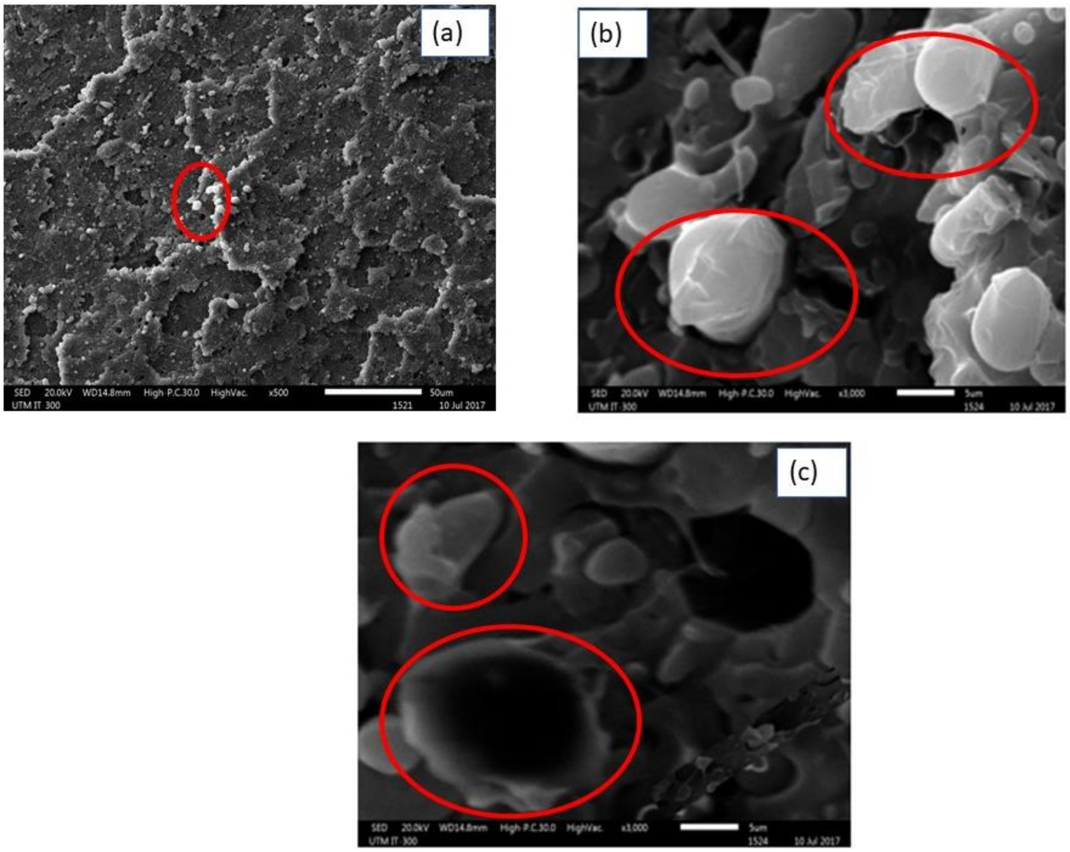
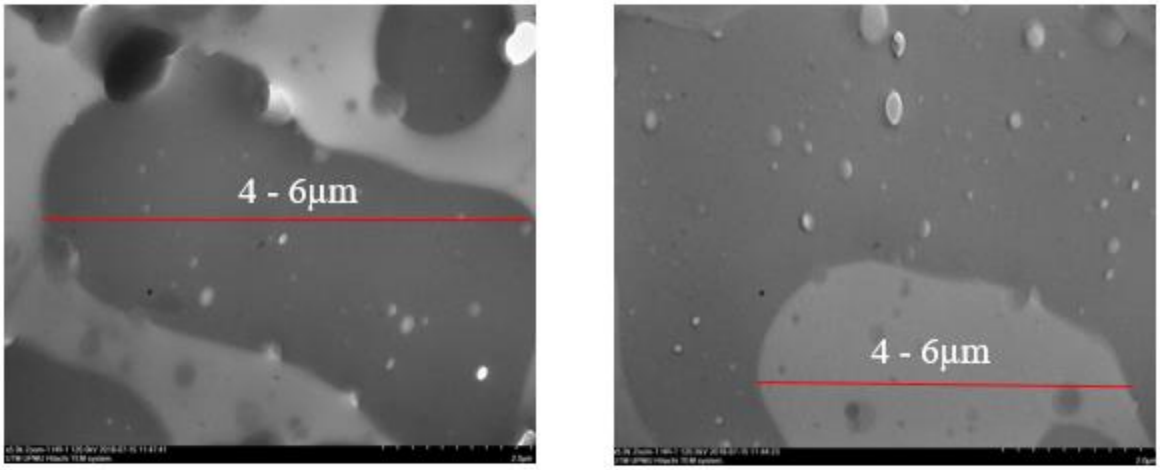


Figure 3. Supermorphology of Uncompatibilzed 50PLA/50PC blend (a) Low magnification micrograph of PC (b) high magnification of clustered PC domains (c) pull-out analysis due to fracture

TEM Analysis

The incorporation of CA- TBATPB/TA-2022 eradicates the large spaces between the PLA domains and the PC matrix. It is observed from Figure 4 that only slight separation boundaries were noticeable between the two phases. The PLA/PC blend formation could be observed from the development of a new interlayer between the PLA and the PC matrix. The PC droplets appeared larger and flatter as compared to physical blends, showing the size of about 4-6 µm (indicated by red line). This observation can be described by a reduction in the surface tension of the PC due to the creation of the PLA-PC copolymer [27].



(a) 50PLA/50PC/0.2CA/5TA-2022 (b) 50PLA/50PC/0.2CA/5TA-1578

Figure 4. TEM micrographs of impact-fractured surface of PLA/PC blends with triacetin and (a & b) catalyst

Thermogravimetric Analysis

TGA and DTG thermograms are depicted in Figures (5) and (6). The thermal degradation of PLA undergoes a single step degradation having initial degradation temperature (T_i) at approximately 283 °C and final degradation temperature (T_f) of about 380 °C. Additionally, maximum degradation temperature (T_{max}) occurs at 364 °C as illustrated in Figure (5) [21].

Cinelli *et al*. [4] reported that PC represented a two-stage decomposition temperature behaviour in air; T_i and T_f which is approximately 434 °C and 544 °C, respectively with a T_{max} of about 522 °C. However, a shoulder peak was noticed before T_i around 430 °C that represents chemical reactions associated with the thermal oxidative degradation along with chain scission of isopropylidene bond. Additionally, it also reflects the molecular linkages between oxygen atom where cross linking and branching reactions take place [22–23].

50PLA/50PC.blends without and with compatibilizers/catalyst exhibited a two-stage degradation process. The first degradation process is associated with PLA, whereas the second one is related to PC. T_i of the blends with and without catalyst/compatibilizers begin at a temperature associated with the PLA; whereas, it shows a T_f around 450 °C – 520 °C that is before the end point temperature related to PC. Furthermore, it is clearly visible that the T_{max} of all the blends as depicted in Figure (6) is quite similar with PLA rather than PC. Hence, the T_{max} of PC in the blends is changed to that of pure polymer and measured around 355 °C – 380 °C. These findings may be possibly due to the degradation of PLA which in turn also results in the degradation of PC in the blends. From Figure (6), it is noticeable that the blends have a broader peak as compared to PLA, thereby increasing the thermal resistance of blends in the presence of PC [28].

Table 3. Thermogravimetric analysis of PLA, PC and its different types

Sample Name	T_i (°C)	T_{max} (°C)	T_f (°C)	Ash content (%)
PLA	283	364	380	2.6
50PLA/50PC	320	361	444	14
50PLA/50PC/5TA-2022	301	346	488	14
50PLA/50PC/5TA-1578	321	357	481	12
50PLA/50PC/0.2CA	321	365	445	12
50PLA/50PC/0.2CA/5TA-2022	313	350	487	14
50PLA/50PC/0.2CA/5TA-1578	301	341	485	12
PC	434	522	544	30

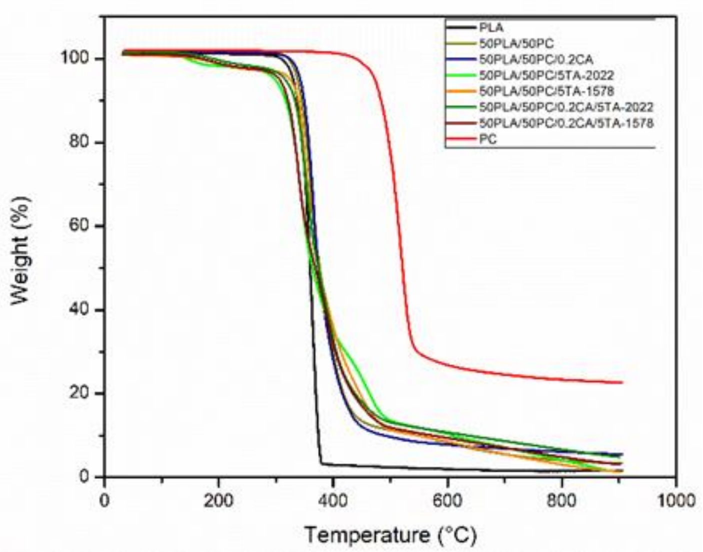


Figure 5. Thermogravimetric analysis of PLA, PC and its different types

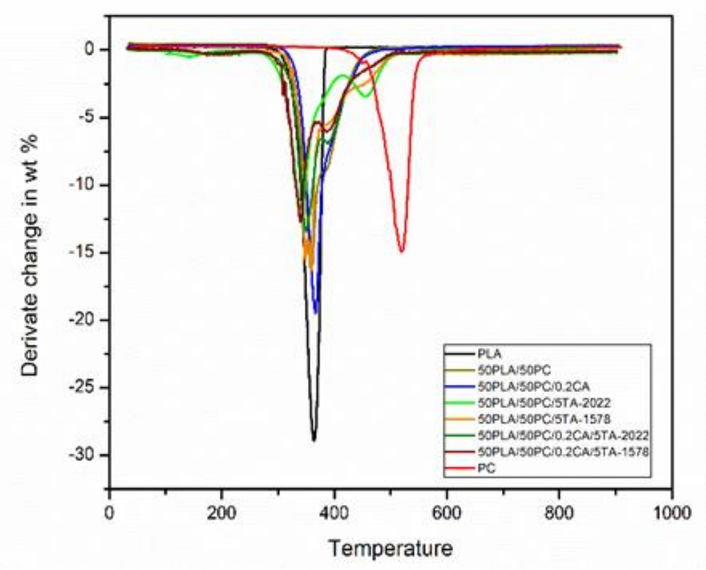


Figure 6. DTG analysis of PLA, PC and its different types

Dynamic Mechanical Analysis

DMA thermograms of storage modulus E' and tan δ are depicted in Figures (7) and (8). PLA exhibited a tan δ peak at 69 °C in Figure (8). Normally, the tan δ peak indicates the relaxation peak of a polymer which is related to T_g. Furthermore, 50PLA/50PC blend show two T_g peaks around 72 °C and 165 °C. The two peaks are indicative of T_g of PLA and PC within the blend, respectively. Hence, the presence of separated PC and PLA phases within the blends can be clearly witnessed [25].

The tan δ peak height represents the amorphous segments mobility within the polymer. It can be clearly seen in the 50PLA/50PC tan δ peak that the addition of PC broadens the peak at 72 °C, which is associated with PLA, thereby having a reduced height. However, the peak related to PC at 165 °C is as sharpened and heightened, indicating its increased mobility [28,33].

In the presence of both the compatibilizer (TA-2022 and TA-1578), the blends exhibited similar T_g ~65 °C – 69 °C as associated with PLA; in addition to another peak as depicted around ~ 127 – 129 °C which is less than that of PC. These results corroborated the strong adhesion at PLA-PC interface [29].

When considering Figure (7), it is noticeable that the E' shows a minimum value ~ 81 °C above its T_g value. Therefore, the E' shows a minimum value ~ 122 °C which signifies that PLA phase crystallization proceeded during the low scan rate of DMA run. However, the decrease in the E' of PLA in the PLA/PC blends starts at lower temperatures because of the plasticization effect of compatibilizer (TA-2022/TA-1578). Furthermore, it is evident that less decrease in E' is observed in the presence of CA/TA in PLA/PC blends. This confirms the formation of chemical links between PLA and PC; thereby hindering the chain mobility and crystallization. Hence, this increases the thermal resistance of PLA where it can withstand its mechanical properties up to ~ 110 °C, which is higher than T_g of typical PLA and lower than the T_g of typical PC [30,31].

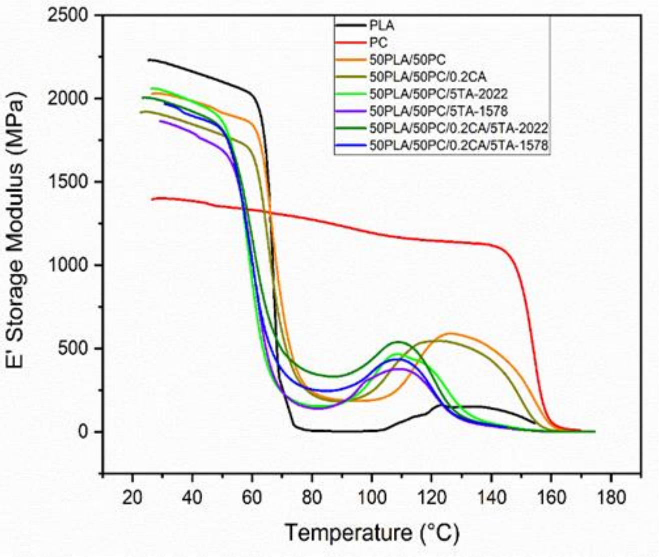


Figure 7. Storage Modulus of PLA, PC and its different types

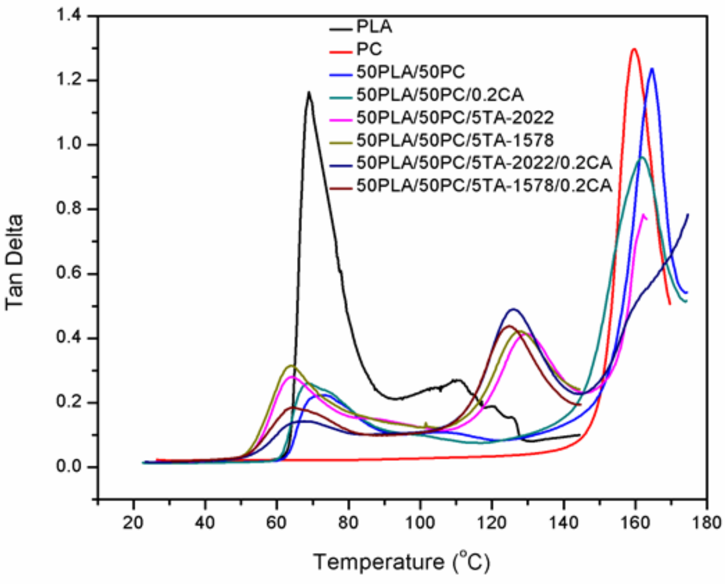


Figure 8. Tan Delta of PLA, PC and its different types

Conclusions

The mechanical analysis of PLA/PC blends showed that the elongation of the blend improved as the PC content increased in PLA and the Young's modulus decreased. The optimum elongation at break obtained at 50 wt% of PC in the PLA-PC copolymer, allowing the attainment of a continuous phase domains. Addition of the catalyst and triacetin increased the Young's modulus due to the enhancement of compatibility and polymer scission in the PLA domains. Therefore, at lower PC contents, a co-continuous phase morphology is achieved. The improvement in elongation at break shows that TA-2022 is more effective than TA-1578 triacetion.

The morphological FESEM and TEM analyses showed the properties of CA-TBATPB/TA on the phase separation of the copolymer. As a result of PLA chain scission, the interfacial adhesion between PLA and PC was improved, and the measurement of the discrete PLA domains augmented in arrangement to co-continuous phase morphology.

DMA analysis depicted an increase of thermal resistance of PLA where it can withstand its mechanical properties up to ~ 110 °C. DMA analysis clarified that the increased PC content in 50PLA/50PC/0.2CA/5TA-2022 reduced the crystalline fraction thereby reducing the mobility of PLA domains. The mechanical stability of PLA/PC blend is higher than T_g of typical PLA. This effect was probably due to the links created between the PLA and PC.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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