

# **TOUGHNESS MODIFICATION OF PLA BIOCOMPOSITES**

Teja Pešl, Silvester Bolka, Tamara Rozman, Rebeka Lorber, Rajko  
Bobovnik, Blaž Nardin

Faculty of Polymer Technology, Slovenj Gradec, Slovenia

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doi: 10.5281/zenodo.5336135  
teja.pesl@ftpo.eu

**Abstract:** *High brittleness of polylactic acid (PLA) is the main drawback for the use of this material in the packaging sector. To overcome this drawback, there are many PLA blends with other thermoplastic tough materials. To maintain the biodegradable properties of PLA blends, the preparation of blends with other biodegradable materials is preferred. The most commonly used are poly(3-hydroxybutyrate) (PHB) and polybutylene adipate terephthalate (PBAT). The disadvantage of PHB is its high price, and the disadvantage of PBAT is the incompatibility of PLA and PBAT. For PLA-PBAT blends, multifunctional reactive polymers are used as reactive compatibilizers. For packaging applications, epoxy oligomers can be used as reactive compatibilizers. To reduce the price of PLA blend, biomass can be added as filler. In the present work, the PLA matrix was mixed with 15 wt.% and 20 wt.% PBAT in the presence of a multifunctional reactive polymer with epoxy groups. As biomass, 5 wt.% and 10 wt.% of hops were added along with compatibilizer. The mechanical, thermal and impact properties were tested and evaluated. The presence of hops keeps the thermal properties of biocomposites at the level of pure PLA. The addition of PBAT lowers the stiffness and strength, but dramatically increases the toughness of the biocomposites. In biocomposites, cold crystallization of PLA occurs at lower temperatures compared to neat PLA. From the results, we can conclude that hops increases the stiffness and decreases the glass transition temperature, while the addition of PBAT decreases the stiffness and glass transition temperatures, but both together act as nucleating agents for the crystallization of PLA matrix. Reactive compatibilization was successful as the biocomposite exhibited over 200 % higher impact strength and over 240 % higher elongation*

*at break compared to the neat PLA matrix. The biocomposites are cheaper alternative and can be used in the packaging sector due to their good stiffness, strength, toughness and low price compared to the petroleum-based materials currently used in the packaging sector. Another advantage is that biomass can be used as a filler in biocomposites without the need for surface treatment as a suitable compatibilizer is used.*

**Keywords:** PLA, PBAT, toughness modification

## 1 INTRODUCTION

High-volume applications of bio-based and biodegradable materials are driven by low carbon footprint, renewability, and compostability. Like other fossil-based thermoplastic materials, bio-based and biodegradable materials have some drawbacks. These can be avoided by blending these materials with other thermoplastic materials. Polylactic acid (PLA) and poly(butylene adipate-co-terephthalate) (PBAT) are polyester materials that have attracted a lot of attention due to their biodegradability. PLA is synthesized from renewable resources, while PBAT is petroleum-based. PLA is a semi-crystalline polymer with a melting point around 180 °C and a glass transition temperature of 50 °C to 65 °C. The thermal properties and toughness of PLA are insufficient in many applications, including packaging applications (Reddy et al., 2013). To improve the poor performance of PLA, blends can be made with flexible but biodegradable materials such as polycaprolactone (PCL), poly(propylene carbonate) (PPC), poly(butylene succinate) (PBS), poly(butylene succinate-co-adipate) (PBSA), and PBAT (Wang et al., 2019). PLA/PBAT blends were prepared using different processing technologies and in a wide range of ratios between 90/10 to 10/90 wt.%. For blown film, the maximum PLA content was 40 wt.%. The limited miscibility between PLA and PBAT was confirmed with two almost unchanged glass transition temperatures in all blends. Two-phase morphologies were observed, droplet-matrix and co-continuous morphology. A blend with 10 wt.% PBAT showed the highest stiffness, and a blend with 90 wt.% PBAT showed the highest elongation at break (Su et al., 2020). When PLA is blended with PBAT, the interpenetrating network can be prepared in the composition range between 10 and 20 wt.% of PBAT and the elongation at break can be increased from 10% to 300% (Deng et al., 2018). The miscibility of PLA/PBAT blends showed partial miscibility of PBAT in the PLA-rich phase at PBAT content less than 1 vol.%. A dependence of the partial miscibility on the molecular weight of PBAT was demonstrated. As the molecular weight of PBAT increased, the partial miscibility decreased (Jalali Dil et al., 2015). To overcome

the limit of miscibility with the amount of PBAT, the use of a suitable compatibilizer is an effective means. When 30 wt.% PBAT was added to PLA, the 10 wt.% PLA grafted with glycidyl methacrylate improved the tensile strength and elongation at break (Lyu et al., 2020). PLA-polyethylene glycol-PLA (3 wt.%) were synthesized as effective compatibilizers with different chain lengths to promote interfacial structure and mechanical properties in PLA/PBAT (70/30 wt.%) blends. In the presence of the compatibilizer, the fine phase morphology appeared due to the increased interfacial adhesion between PLA and PBAT as the compatibilizer moved to the interface of PLA/PBAT due to the high shear during compounding. The PLA-polyethylene glycol-PLA compatibilizer decreased the stiffness and strength of the compounds, the elongation at break was not significantly affected, while the toughness was improved (Ding, Feng, et al., 2018). The addition of tri-block copolymers PLA-PBAT-PLA (5 wt.%) to PLA/PBAT blends (70/30 wt.%) improved the elongation at break, but simultaneously decreased the strength (Ding, Lu, et al., 2018). For electrospinning, the compatibilizer tetrabutyl titanate (2 and 4 wt.%) was used in PLA/PBAT (70/30 wt.%) blends. The compatibilizer resulted in homogeneous bead-free fibers and smoother fiber surface due to improved compatibility of PLA/PBAT blends due to improved interfacial adhesion between PLA and PBAT (Khatsee et al., 2018). By using peroxide as a modifier in a PLA, PBAT and poly(butylene succinate) matrix, a super-tough PLA blend was prepared. The interfacial adhesion between all three components and the reduced particle size of PBAT were the reason for the super-tough material (Wu et al., 2019). The toughness of the PLA/PBAT blend was improved by the addition of triethyl citrate and toluene diisocyanate. The synergistic effect of plasticizer and compatibilizer improved the chain mobility and interfacial interaction between PLA and PBAT. This resulted in improved tensile and impact properties and accelerated cold crystallization behavior (Phetwarotai et al., 2019). The literature reports the use of multifunctionalized epoxy polymers as proper compatibilizers for PLA/PBAT blends (Arruda et al., 2015; Carbonell-Verdu et al., 2018; Lamnawar et al., 2018; Zhao et al., 2020). Multifunctionalized epoxy (0.25 wt.% and 0.5 wt.%) used in the reactive extrusion of PLA/PBAT blends (80/20 wt.%) increased the stiffness and elongation at break due to chain elongation, chain branching and coupling of PLA and PBAT (Al-Itry et al., 2014). Addition of epoxy functionalized poly(lactide) in PLA/PBAT blends for blown film production increased melt strength and showed increased bubble stability. Higher PLA content, up to 70 wt.% PLA, could be achieved with the compatibilizer (Schneider et al., 2016). Biodegradation of PLA/PBAT samples in soil showed an increased degree of crystallinity during biodegradation. Higher crystallinity inhibited

biodegradation. The multifunctional chain extender (1 wt.%) also inhibited biodegradation of blends (Palsikowski et al., 2018).

## 2 MATERIAL AND METHODS

### 2.1 Sample

Commercially available PLA with the trade name Ingeo 4043D was provided by Plastika Trček, Slovenia. Commercially available PBAT with the trade name Ecoflex F Blend C1200 was purchased from BASF, Germany. A commercially available multifunctionalized epoxy polymer with the trade name Joncryl ADR 4468 was purchased from BASF, Netherlands. A commercial TPU copolymer with the trade name Kuramiron U TU -S5265 was purchased from Kuraray, Germany. Commercial hops with the trade name Styrian Aurora in the form of pellets were donated by Slovenian Institute of Hop Research and Brewing, Slovenia. The composition of the samples is shown in Table 1.

*Table 1: Composition of the samples and number of compounding cycles*

<b>Sample</b>	<b>PLA (wt.%)</b>	<b>PBAT (wt.%)</b>	<b>TPU (wt.%)</b>	<b>Joncryl (wt.%)</b>	<b>Hops (wt.%)</b>
PLA	100	0	0	0.0	0
PLA15PBAT10H	70	15	4.5	0.5	10
PLA15PBAT5H	75	15	4.5	0.5	5
PLA20PBAT5H	70	20	4.5	0.5	5

### 2.2 Processing

For the compounding cycle, the materials were mixed separately and extruded on the Labtech LTE 20-44 twin screw extruder. The screws had a diameter of 20 mm, an L/D ratio of 44:1, a screw speed of 300 rpm, and an increasing temperature profile from the hopper (145 °C) to the die (180 °C). After compounding, the two produced filaments were cooled in a water bath and cut into pellets with a length of about 5 mm and a diameter of 3 mm.

Injection molding was performed on Krauss Maffei 50-180 CX with a screw diameter of 30 mm. The temperature profile was increasing from the hopper (165 °C) to the nozzle (180 °C). The mold temperature was set to 30 °C and the cooling time to 20 s. The back pressure was set to 195 bar and the screw speed to 100 rpm. The injection speed was set to 60 mm/s and to 20 mm/s for the last 2 mm.

### 2. 3 Characterization

Flexural and tensile tests were performed on the Shimadzu AG -X plus according to ISO 178 and ISO 527-1, respectively. Five measurements were taken for each specimen. In the tensile tests, tensile stiffness ( $E_t$ ), tensile strength ( $\sigma_m$ ), elongation at yield ( $\epsilon_m$ ) and elongation at break ( $\epsilon_{tb}$ ) were determined. In the flexural tests, the flexural stiffness ( $E_f$ ) and the flexural strength ( $\sigma_fM$ ) and the elongation at yield ( $\epsilon_fM$ ) were evaluated. Thermomechanical properties were investigated using a Perkin Elmer DMA 8000. The specimens were heated at 2 °C/min from 25 °C to 120 °C under air atmosphere. A frequency of 1 Hz and an amplitude of 20  $\mu$ m in dual cantilever mode were used. Thermal measurements were performed using a differential scanning calorimeter (DSC 2, Mettler Toledo) under nitrogen atmosphere (20 mL/min). The temperature of the samples was raised from 25 to 180 °C at a heating rate of 10 °C/min and held in the molten state for 5 min to erase their thermal history. After cooling at 10 °C/min, the samples were reheated to 180 °C at 10 °C/min. The crystallisation temperature ( $T_c$ ), crystallisation enthalpy ( $\Delta H_c$ ), melting temperature ( $T_m$ ), and melting enthalpy ( $\Delta H_m$ ) were determined from the cooling and the second heating scan. Thermogravimetric analyses (TGA) were performed using a Mettler Toledo TGA/DSC 2+ thermal analyzer. Analyses were performed in a nitrogen atmosphere (20 mL/min) from 40 to 600 °C with a heating rate of 10 °C/min, followed by a segment in an oxygen atmosphere (20 mL/min) from 600 to 650 °C using an Al<sub>2</sub>O<sub>3</sub> crucible without a lid. Impact tests were performed using a pendulum Dongguan Liyi Test Equipment, type LY -XJJD5, according to ISO 179. The distance between the supports was 60 mm, and a pendulum with 5 J and 2 J was used for the impact and notched impact tests, respectively.

## 3 RESULTS AND DISCUSSION

### 3. 1 Mechanical properties

As expected, the addition of PBAT to PLA (Table 2, Figure 1) decreased the strength and stiffness of PLA/PBAT blends. The addition of hops lowered the flexural strength but increased the flexural stiffness, lowered the tensile strength and stiffness and the elongation at break. The addition of PBAT drastically increased the elongation at break. We can conclude that the PLA/PBAT biocomposite with hops achieved higher toughness by adding Joncryl and TPU as compatibilizers, which achieved a synergistic effect. The improved toughness is the result of good interfacial bonding between PLA and PBAT matrix and also hops. TPU improved the interfacial bonding between the

thermoplastic matrix and hops, and Joncryl successfully bonded PLA and PBAT. Lower hops content and higher PLA content in the composite had minimal effect on stiffness, increased strength and elongation at break. We can conclude that Joncryl successfully coupled the PLA and PBAT matrix and the compatibilizer TPU increased the interfacial adhesion between hops and thermoplastic matrix. Higher PBAT content and lower hops content decreased the stiffness and strength, but dramatically increased the elongation at break, which can be attributed to the plasticizing effect of PBAT and simultaneously to the coupling of an additional amount of PBAT to PLA as a result of the addition of Joncryl. Lower PLA content and higher PBAT content exhibit lower strength and stiffness, and the elongation at break is greatly increased. Multifunctionalized epoxy polymer successfully coupled PLA and PBAT, probably due to the formation of smaller PBAT domains in the PLA phase (Ding, Feng, et al., 2018).

Table 2: Summarized results from the flexural and tensile tests with standard deviation values

Sample	Flexural test results			Tensile test results			
	$E_f$ (GPa)	$\sigma_{fM}$ (MPa)	$\epsilon_{fM}$ (%)	$E_t$ (GPa)	$\sigma_m$ (MPa)	$\epsilon_m$ (%)	$\epsilon_{tb}$ (%)
PLA	3.38±0.07	105.2±0.8	4.49±0.04	3.17±0.21	71.8±1.0	3.25±0.22	4.81±0.27
PLA15PBAT10H	2.87±0.02	71.5±0.2	3.38±0.01	2.47±0.23	46.9±0.7	2.76±0.05	3.93±0.21
PLA15PBAT5H	2.84±0.01	74.1±0.3	3.50±0.01	2.59±0.17	50.4±0.5	2.94±0.04	8.87±1.16
PLA20PBAT5H	2.48±0.01	64.3±0.2	3.85±0.04	2.45±0.33	42.9±0.8	2.88±0.04	16.50±2.04

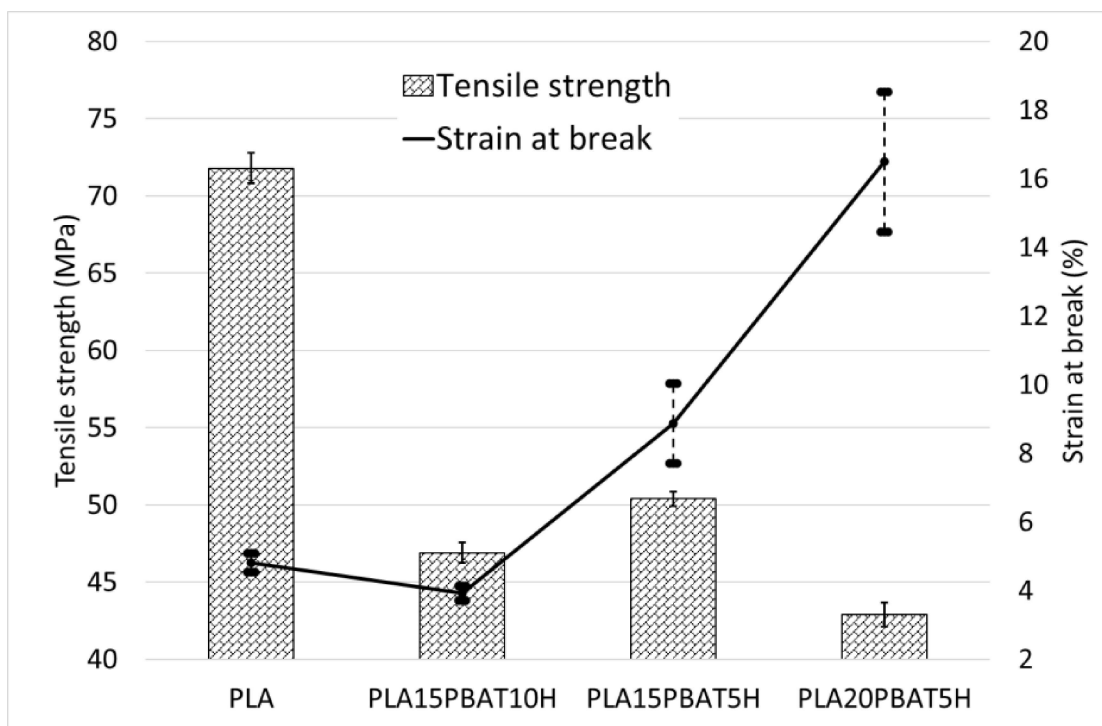


Figure 1: Summarized results of the tensile strength (bars) and strain at break (line)

### *3. 2 Thermo-mechanical properties*

The storage modulus curves (Figure 2) showed the first drop in glass transition temperature for PLA. With increasing PBAT content, the glass transition temperature decreased. A higher percentage of hops decreased the glass transition temperature. The highest storage modulus is exhibited by pure PLA, and the lowest storage modulus is exhibited by the biocomposite with the highest PBAT content (sample PAL10PBAT5H). After glass transition, the neat PLA had the lowest storage modulus and the highest cold crystallization temperature. For the biocomposites, the cold crystallization temperature of PLA was lower. We can conclude that the improved cold crystallization behavior of PLA is due to the addition of multifunctionalized epoxy polymer. The highest storage modulus was exhibited by the samples with the highest hops content. Most likely, hops acted as nuclei for the propagation of cold crystallization at lower temperatures, as the smaller size of PBAT particles was less sterically hindering PLA crystallization. Two peaks are observed in the loss modulus curves (Figure 3) at about 60-65 °C and 95-110 °C, corresponding to the glass transition of PLA and cold crystallization of PLA, respectively. As the amount of PBAT increased, the height of the peak for the glass transition of PLA decreased. With the addition of PBAT, the peaks for the glass transition of PLA shifted to a lower temperature that was not affected by the amount of PBAT. With a higher amount of hops, the peak height of the loss modulus for the glass transition of PLA remained at the same level, but the peak is shifted to lower temperatures. The shift of the peaks of glass transition of PLA to lower temperatures illustrates the improved miscibility of PLA and PBAT at the interface. The higher loss modulus at lower temperatures for the biocomposites compared to neat PLA also demonstrates the plasticizing effect of PBAT in the biocomposites. The most pronounced shift in peak position to lower temperatures for the biocomposites compared to neat PLA is seen in the loss factor curves (Figure 4). The most elastic behavior in the glass transition region of PLA was exhibited by the sample with the highest PBAT content (PLA20PBAT5H). A higher addition of hops acted as a plasticizer and resulted in better damping behavior, as shown by a higher peak of loss factor compared to a lower hops content and the same PBAT content. The peak of loss factor is slightly shifted to lower temperature at higher hops content. We can conclude that hops as an additive to Joncryl improves the miscibility of PLA and PBAT, which is most likely due to the addition of TPU leading to good interfacial interactions between PLA and PBAT and hops.

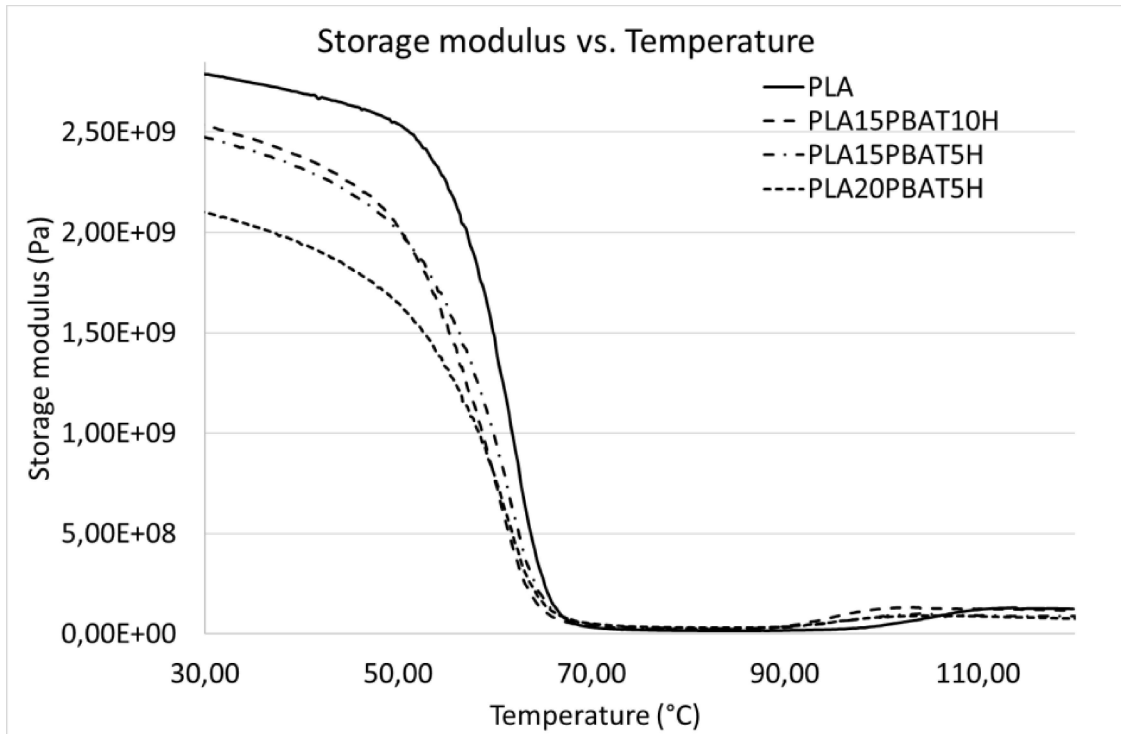


Figure 2: Summarized results of storage modulus

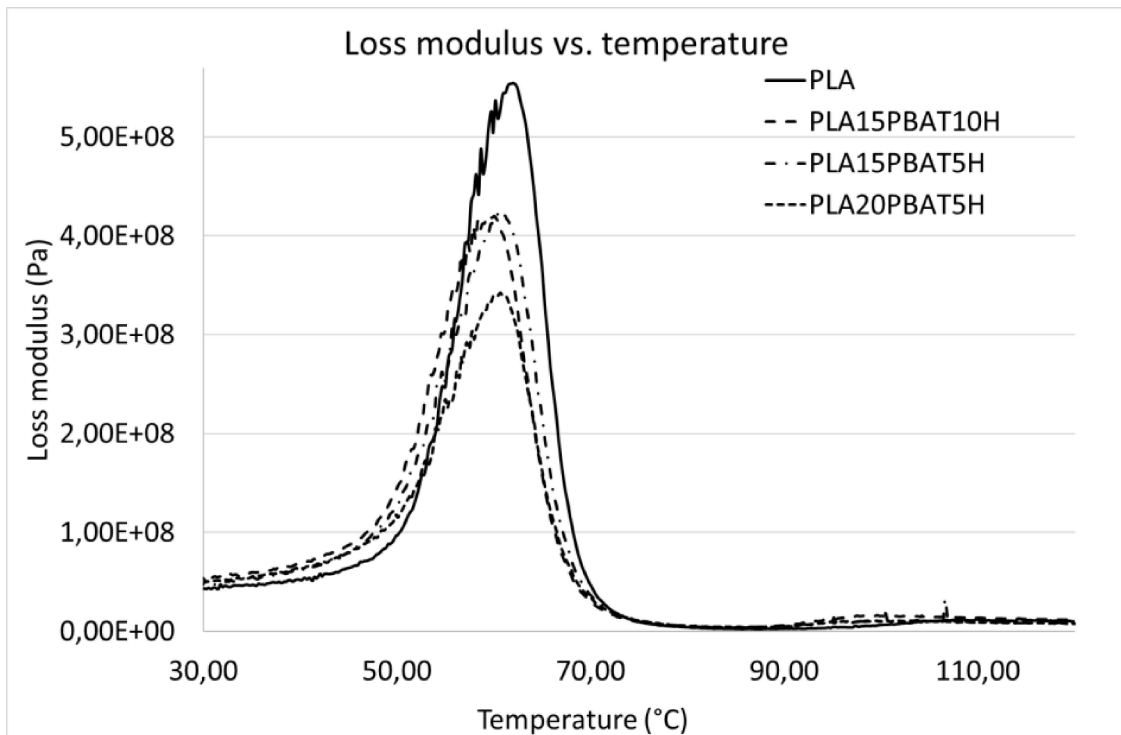


Figure 3: Summarized results of loss modulus



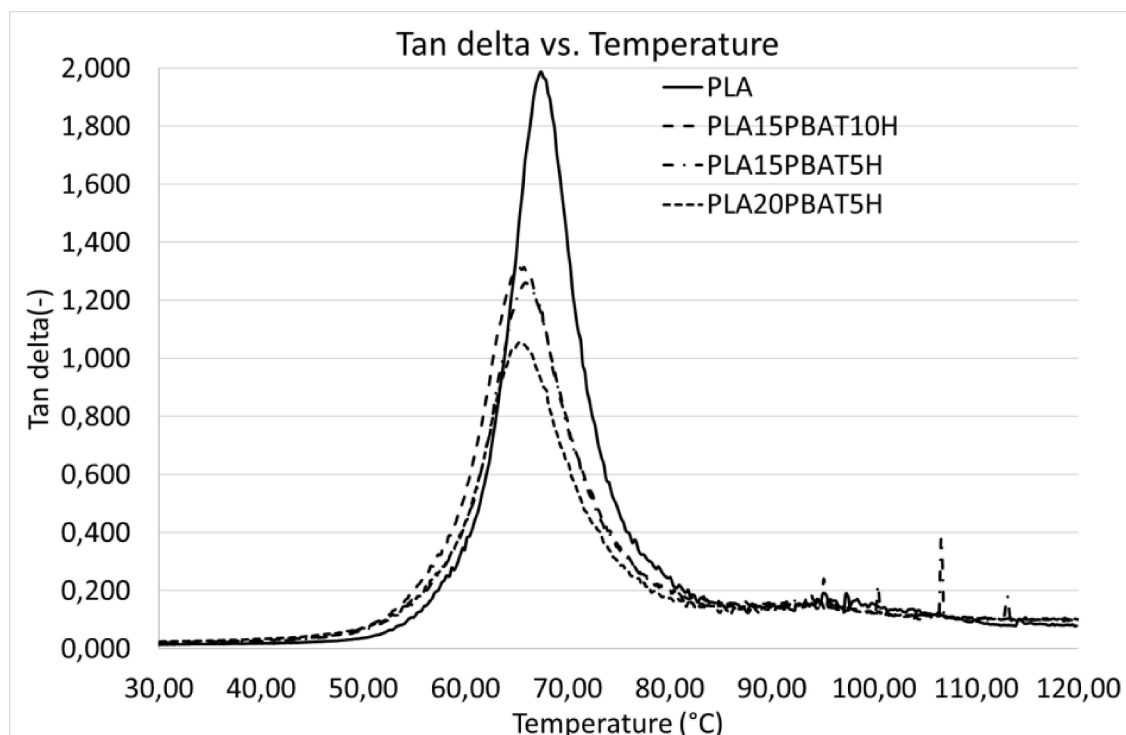


Figure 4: Summarized results of loss factor

### 3. 3 Thermal properties

Due to the low crystallization of neat PLA, cold crystallization occurred during the second heating, and no crystallization was observed during cooling (Tables 3 and 4). The addition of PBAT to PLA resulted in an overlap of melting of PBAT and cold crystallization of PLA upon heating. With a higher amount of PBAT in PLA/PBAT blends, the peak height for cold crystallization of PLA and melting decreased. The glass transition temperature shifted to lower temperatures, and the cold crystallization temperature and melting temperature shifted to higher values. On cooling, crystallization of PLA occurred (from 69 °C to 71 °C), followed by glass transition temperature and crystallization of PBAT (from 40 °C to 44 °C). A higher amount of hops and a larger amount of PBAT shifted the glass transition temperature of PLA to lower values, which confirmed the DMA results due to the plasticizing effect of hops and PBAT and the formation of good interfacial interactions between PLA and PBAT. The interfacial interactions between PLA and PBAT and the good compatibilization of hops with the thermoplastic matrix suppress the cold crystallization temperature to higher values. Also, the shift of melting temperature of PLA to higher values confirmed good interfacial interactions between PLA and PBAT and the formation of smaller PBAT particles within PLA phase. The conclusion could be that smaller PBAT particles in lower amount can hinder PLA crystallization and larger crystalline PLA domains can be formed. The incorporation of PBAT, hops, Joncryl, and TPU stimulates the crystallization of PLA upon cooling, albeit to a

limited extent. The crystalline PLA domains subsequently inhibit the crystallization of PBAT at lower temperatures. The influence of crystallization kinetics of PLA on crystallization of PBAT could be further explored, and proper morphology of PLA with controlled cooling could lead to even higher toughness of PLA-based blends.

Table 3: Summarized results from the 2<sup>nd</sup> heating from DSC tests

Sample	$T_g$ (°C)	$T_{cc}$ (°C)	$\Delta H_{cc}$ (J/g)	$T_m$ (°C)	$\Delta H_m$ (J/g)	Diff. $\Delta H_m$ (J/g)
PLA	59.7	118.5	13.3	152.5	13.5	0.2
PLA15PBAT10H	58.7	126.8	7.5	153.7	8.1	0.6
PLA15PBAT5H	59.5	129.7	4.8	154.0	5.1	0.3
PLA20PBAT5H	58.7	131.2	1.8	154.1	2.7	0.9

Table 4: Summarized results from the cooling from DSC tests

Sample	$T_g$ (°C)	$T_{c-PLA}$ (°C)	$\Delta H_{c-PLA}$ (J/g)	$T_{c-PBAT}$ (°C)
PLA	54.6	-	-	-
PLA15PBAT10H	-	69.3	0.5	40.3
PLA15PBAT5H	-	70.2	0.2	44.1
PLA20PBAT5H	-	71.3	0.8	40.3

### 3. 4 Impact properties

Biocomposites with the highest PBAT content exhibited the highest impact and notched impact strength. With increasing PBAT content, the impact and notched impact strength increased. Higher toughness could be achieved due to good interfacial interaction between PLA, PBAT and hops. Higher notched impact strength could be achieved due to the plasticizing effect of PBAT, which prevented crack propagation. Despite the good compatibility of hops in PLA/PBAT blends, the proportion of 10 wt.% caused a drop in notched impact strength due to the absence of the plasticizing regions for crack propagation by the hops particles. The size of the hops particles were too large and allowed crack propagation through the particles. The results of the notched impact strength were also confirmed by the tensile tests and the dynamic mechanical tests.

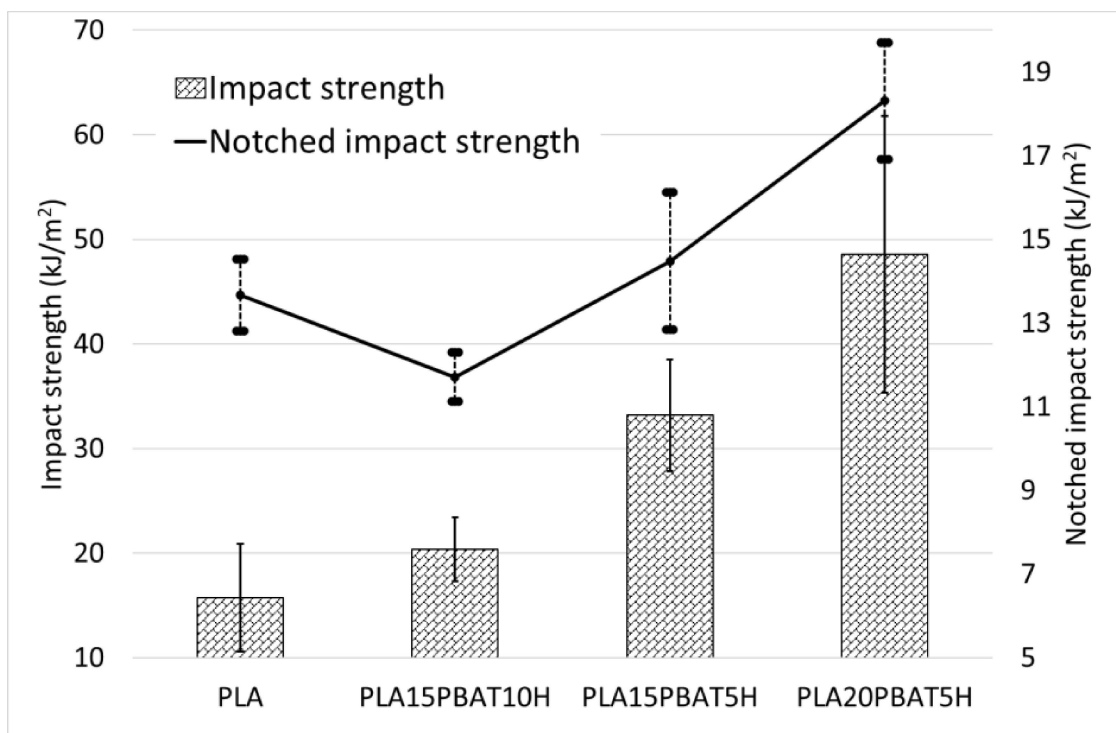


Figure 5: Summarized results of toughness: impact strength (bars) and notched impact strength (line)

#### 4 CONCLUSION

The effect of adding PBAT in two different amounts to PLA and adding hops in two different amounts on the toughness of biocomposites was investigated. In addition, the synergistic effect of the multifunctionalized epoxy polymer and TPU as compatibilizer was investigated and explored.

The synergistic effect of multifunctionalized epoxy polymer and TPU copolymer as compatibilizer leads to new properties of PLA-based thermoplastic materials. The toughness was drastically increased, the composites remained biodegradable, and the processing temperature could be lower compared to neat PLA. An interesting correlation was described between the crystallization kinetics of PLA and the crystallization of PBAT. The biomass could be an effective additive for nucleation and at the same time a plasticizer for PLA-based thermoplastic composites. To avoid immiscibility of PLA and PBAT, the multifunctionalized epoxy polymer together with the compatibilizer TPU copolymer achieved excellent interfacial interaction between PLA and PBAT and at the same time enabled good compatibilization of biomass without any surface pretreatment of biomass. The research work described showed a huge improvement in toughness without the need to use other processes besides compounding all materials in one step. Toughness improvement is important in the packaging sector, especially for flexible

packaging. The second important point in the composite material described is the addition of biomass, which both changes the properties and lowers the price of the material. The lower price is often the most important factor in material selection.

The correlation between the PLA crystallization kinetics and the crystallization of PBAT, as well as the effects on the toughness of the PLA/PBAT blends, should be investigated in further research.

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