SEMARAK ILMU
PUBLISHING
202103268166(003316878-P)

Journal of Advanced Research in Applied
Sciences and Engineering Technology

Journal homepage:
https://semarakilmu.com.my/journals/index.php/applied_sciences_eng_tech/index
ISSN: 2462-1943

The Effect of Palm Stearin in Poly (Butylene Adipate-Co-Terephthalate) (PBAT)/ Poly (Lactic Acid) (PLA) Biodegradable Feedstock

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ARTICLE INFO

Article history:
Received 25 May 2023
Received in revised form 22 September 2023
Accepted 5 October 2023
Available online 19 October 2023

Keywords:
PBAT; PLA; palm stearin; composite; compatibilizer

ABSTRACT

Because of environmental concerns, the need to minimize reliance on fossil-based polymers and achieve sustainable growth is important today. In this study, poly (butylene adipate-co-terephthalate) (PBAT) and poly (lactic acid) (PLA) blend at 50/50 wt.% ratio were mixed with palm stearin (PS) ranging from 5 to 20 wt.%. The characterizations of the tri-component PBAT/PLA/PS polymer blend were done based on torque analysis, rheology, thermal properties, and mechanical properties. Results showed the PS induced the compatibilization to PBAT/PLA blend, with the evidence of both T_g PLA and PBAT shifter closer to each other. Moreover, the addition of the PS in the PBAT/PLA matrix decreased the torque value from 4 Nm to 2 Nm of the PBAT/PLA blends and reduce fluidity and showed good thermal stability properties, suggesting that the PS is an alternative material to compatibilized the PBAT/PLA blend. Evidently addition of 15 wt.% PS has also improved the PBAT/PLA blends properties such the elongation at break to approximately 200%.

1. Introduction

The development of environmentally friendly biodegradable polymer materials for disposable plastic items has received a lot of attention in recent years, driven by the desire to minimize the quantity of plastic waste that accumulates in the environment [19]. Biodegradable or bio-based polymers made from renewable resources have received a lot of attention in recent years, not only because of environmental issues and sustainability associated with traditional petroleum-based polymers but also because of their unique applications, particularly in packaging [12] and biomedical devices [9]. Regarding of its resilience, flexibility, and toughness, the polybutylene adipate terephthalate (PBAT) is often considered the best biodegradable polymer for refractory plastics like low density polyethylene [19]. PBAT improves ductility, toughness, and melt strength. Solid particles

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https://doi.org/10.37934/araset.33.1.208218

in polymer blends can aid in achieving a toughness-stiffness balance [8]. Because of these qualities, PBAT is employed in packing materials and agricultural mulch films [18]. However, the polymer has significant drawbacks that restrict its usefulness for injection molding (such as high viscosity, low processability, and poor crystallization ability). To compensate for these drawbacks, PBAT is frequently combined with poly (lactic acid) (PLA) to improve its processability and expand its range of applications.

Based on previous research, poly (lactic acid) (PLA)/poly (butylene adipate-co-terephthalate) (PBAT) is one of the most promising biodegradable polymer blends. PBAT enables PLA to have improved ductility, toughness, melt strength, and processability. On the other side, while PBAT addition to PLA may enhance several characteristics, demonstrated excellent physico-chemical and mechanical properties that being employed for different applications [8,14]. Unfortunately, poor compatibility between PBAT and PLA negatively impacted the mechanical properties of the blends, thus required further modifications [16].

Some studies have investigated the addition of a compatibilizer in the PLA/PBAT blends to improve the compatibility of the PBAT/PLA composites [3,5]. In other studies, particles including silica, graphene and cellulose nanocrystals were added to increase the matrix viscosity that facilitates phase dispersion and thus exhibit better rheology, thermal and mechanical properties [6,8,11]. As the development of the PBAT/PLA composite with broad universal performance gained traction, a new biodegradable component, palm stearin is proposed to be added in the polybutylene adipate terephthalate (PBAT) and poly (lactic acid) (PLA) blends via melt blending technique. Palm stearin is a solid portion of palm oil obtained by partial crystallization at a regulated temperature. It has more varied physical features than palm olein (the liquid portion of palm oil) due to its more variable composition, particularly in terms of solid fat concentration [10,17]. Since, palm stearin (PS) is commercially available and more affordable compared to other biopolymers. Therefore, the main objective of this study is to study the characteristics of the tricomponent poly (butylene adipate-co-terephthalate) (PBAT), poly (lactic acid) (PLA) and palm stearin (PS) polymer blend. The study was accomplished using the torque analysis, differential scanning calorimetry analysis (DSC), thermogravimetric analysis (TGA) and tensile analysis.

2. Methodology

2.1 Material

RBD Hydrogenated Palm Stearin (PS) was acquired from Lipidchem Sdn. Bhd. Poly (butylene adipate-co-terephthalate) (PBAT), grade 801T and a melt flow rate (MFR) of 3-5 g 10 min^{-1} (190 °C, 2.16 kg) with a specific gravity of 1.22 g cm^{-3} were from Shanghai Hengsi New Material Sciences & Technology CO.LTD. A commercial grade poly (lactic acid) pellets was used. All three components were used without any chemical treatment for the preparation of the melt blending. The PBAT and PLA were dried for 24 hours at a temperature of 70 ℃.

2.2 Formulation and Compounding of PBAT/PLA/PS

The method for compounding of PBAT/PLA/PS is known as melt blending by using Thermo Scientific (Haake Rheomix). To avoid the humidity issues during the melt blending, both PBAT and PLA were dried in an oven at 70 °C for 24 hours prior to the mixing process. Then, the 50 wt.% PBAT and 50 wt.% PLA blends were further added with PS at difference percentage as shown in Table 1. Each of the formulation were mixed in the Haake Rheomix internal mixer operated at 60 rpm. The temperature was set at 180 °C, and mixing time was 10 minutes. The process signals are sent to a data acquisition system that permits the recording of the torque and the temperature of heated zones as function of mixing time.

Table 1
Composition of PBAT, PLA and PS in the blends

Formulation	PBAT (wt.%)	PLA (wt.%)	PS (wt.%) phr
50PBAT/PLA/0PS	50	50	0
50PBAT/PLA/5PS	50	50	5
50PBAT/PLA/10PS	50	50	10
50PBAT/PLA/15PS	50	50	15
50PBAT/PLA/20PS	50	50	20

Next, the mixture of poly (butylene adipate-co-terephthalate) (PBAT), poly (lactic acid) (PLA) and palm stearin (PS) were crushed into a smaller particle size. The crushed PBAT/PLA/PS feedstocks were analysed for melting temperature using differential scanning calorimetry (DSC; Star^e system, Mettler Toledo). The test was conducted by weighted and approximately of 7-8 mg of sample and placed on an aluminium crucible and heated up from room temperature to 300 °C at a heating rate of 10 °C/min to determine the melting temperature, T_m and glass transition temperature, T_g in a flowing nitrogen atmosphere. Thermogravimetric analysis (TGA; SDTA581e, Mettler Toledo) was used to obtain the thermal decomposition pattern heated from room temperature to 600 °C at a heating rate of 10 °C/min under nitrogen gas flowing. Then, the feedstock underwent a rheological analysis using a capillary rheometer (Model: RH2000) to measure the viscosity and flow behaviour of the feedstock at different temperatures of 170 °C and 175 °C. Pressure was set at 0.1 MPa with a maximum shear rate of 10, 000 s^{-1}. The rheology was used to identify the flowability property of the blend. Lastly, the PBAT/PLA/PS were shaped into dumbbell-shaped samples by using a hot press machine (Model: ComeTech) at a temperature of 175 °C for preheating within 10 minutes and, compressed and cooled under 5 MPa pressure at 50 °C for 10 minutes. Cooling water flow was used for faster cooling effect. All samples were kept in a desiccator prior to analysis. A Universal Tensile Machine Tinius Olson (Model: $H_{50}KT$A) was used to test the tensile strength of the PBAT/PLA/PS blend (5 specimens) in accordance with the ASTM D882. The load cell used was 2 kN with ±0.5 percent accuracy and the crosshead speed of 2 mm/min.

3. Results and Discussion

3.1 Torque Analysis

Figure 1 shows the changes in torque as a function of mixing time at 60 rpm and 180 °C. The mixing torque, proportional to the shear stress of the mixer indicates the work energy is consumed to disperse and distribute the polymer components within the blend. The mixing time is defined as the time required for a mix to reach steady state. It clearly showed that the torque value decreased as the addition of PS content in the formulation was increased. This may be explained by the fact that lesser friction occurred when more PS was added, contributing to a decreased viscosity of the mixture. This result agrees with Omar *et. al.,* [15] who observed a similar trend where the effect of a larger concentration of smaller molecular weight component of PS in a PS/PE binder formulation of 316L SS resulted in low viscosities and low torque value.

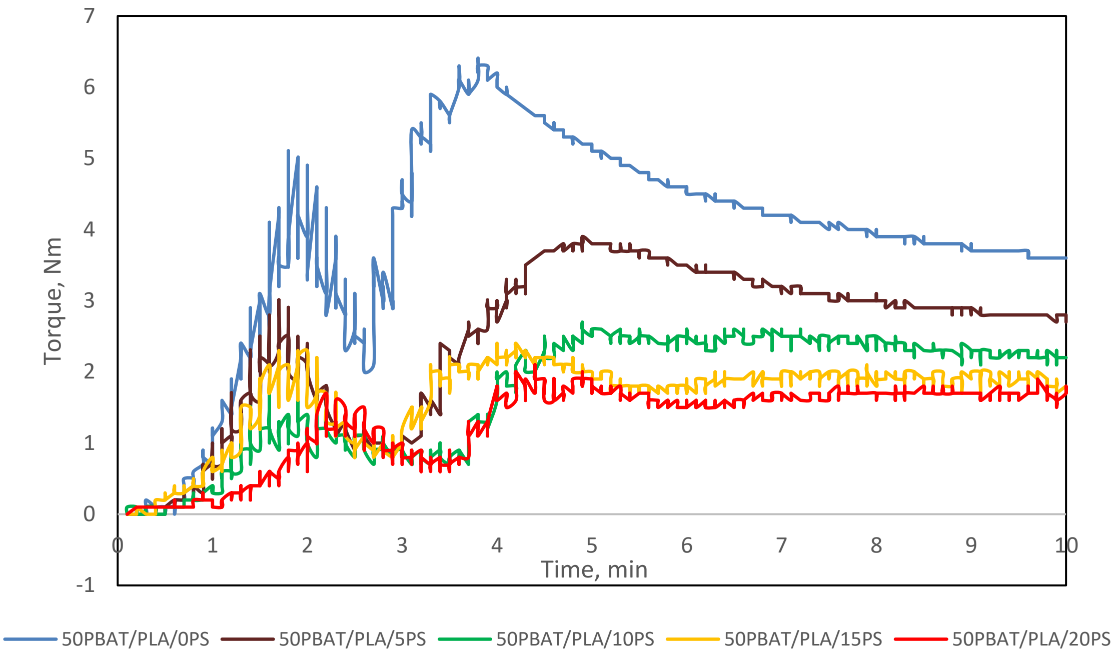


Fig. 1. Mixing torque as a function of mixing time 60 rpm at 180 °C

Figure 1 also shows that all torque value stabilizes after 4 to 5 minutes, indicating a uniform mixing. The results show that the mixing of feedstock 50PBAT/PLA/0PS without PS has stabilized at approximately 4 Nm torque value. The torque value decrease to 3.0 Nm with addition of 5% PS. Furthermore, by using 20% PS in the PBAT/PLA blends, a more uniform feedstock was created, and the torque achieved a steady state value in only 4 minutes. It was also observed that low torque value of 1.7 Nm was achieved for feedstock 50PBAT/PLA/20PS, indicating the optimum composition of the PS in the blend.

3.2 Rheology Analysis

The graph of viscosity versus shear rate for different PS content in the PBAT/PLA/PS formulations are shown in Figure 2. As the pressure was increased, the viscosity decreased for all feedstock formulations, exhibiting a pseudoplastic behaviour that is suitable for injection moulding application. Interestingly, at 175 °C, adding up to 20% PS caused resistance to PBAT/PLA mix flowability, increasing shear viscosity in the region of shear rate 0-4000 s^{-1}. Hence, the PBAT/PLA blends suffers a large shear action with added PS as it flown through the capillary and resulted in higher shear rate. However, the shear viscosity slightly decreased with addition of PS for the shear rate when it increased from 4000 to 10,000 s^{-1} as show in Figure 2. This result agrees with the torque value analysis. As stated before, the viscosity was reduced with an increased PS content in the polymer blends. In other words, PS had functioned as a modifier to reduce the viscosity of the PBAT/PLA mixture.

Since the feedstock exhibits a pseudoplastic behaviour, it should obey Eq. (1) in determining the value of shear sensitivity of *n*.

$$\eta = K\gamma^{n-1} \tag{1}$$

where η is viscosity, γ is shear rate, *k* is coefficient and *n* is shear strain sensitive exponent which is less than 1. The lower the value of *n*, the more sensitive the viscosity is to shear rate. In turn, the higher the value of *n*, low sensitivity of viscosity changes with shear rate that considered from many researchers [1,2,7,15]. The value of *n* for each feedstock was determined to be less than 1 as shown in Table 2, which indicates a pseudoplastic behaviour and suitability for moulding process. High value of *n* with addition of PS in the blends shows a better homogeneity of the feedstock.

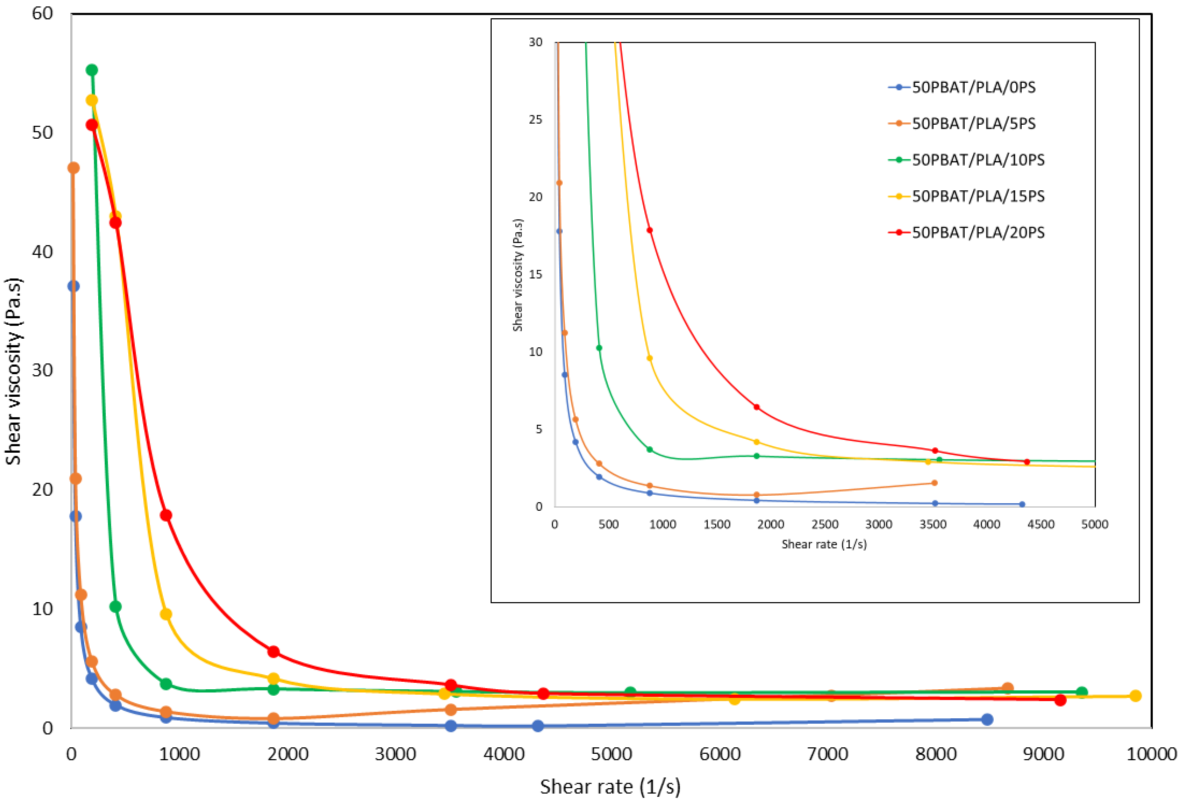


Fig. 2. Shear viscosities of PBAT/PLA/PS at 175 °C

From Table 2, it shows that at temperature 175 °C, the data listed were in the allowable range of pseudo-plastic compared at temperature 170 °C. 170 °C were not suitable use for the next hot press process. This is because the shear sensitivity index (*n*) value in the pseudo-plastic flow area ranges from 0.4 to 0.7. As a result, the feedstock's viscosity varies with shear rate more quickly with the lower value of the shear sensitivity index (*n*).

Table 2
Shear sensitivity, (*n*)

Temperature	170 °C	175 °C
50PBAT/50PLA/0PS	0.16	0.161
50PBAT/50PLA/5PS	0.04	0.511
50PBAT/50PLA/10PS	0.94	0.433
50PBAT/50PLA/15PS	0	0.455
50PBAT/50PLA/20PS	0.12	0.448

The rheological behaviour explicitly shows how important the development of an optimized feedstock composition is. Even though rheological test has been carried out, it is difficult to translate the data during injection moulding practice. This is because the arbitrary value must depend on machine pressure and sprue, gate, and mould design to make it possible for higher viscosity to be tolerated. However, the data would give some insights on the scope of the parameter condition to be applied during injection moulding practice. Initial experiments on rheological behaviour showed that the choice of a suitable composition of PS in PBAT/PLA blends is a major challenge.

3.3 Differential Scanning Calorimetry (DSC)

The melting temperature, T_m and glass transition temperature, T_g of the PLA, PBAT and the co-50PBAT/PLA blend that were measured using differential scanning calorimetry (DSC) are shown in Figure 3. T_g of the PBAT and PLA are observed at 44.9 °C and 60.1 °C, respectively. While the co-50PBAT/PLA blend curve reveals two inflection points for the PLA and PBAT chains. With the presence of PLA, the T_g of PBAT rises to 45.7 °C, which could be attributed to the PLA chains in the co-50PBAT/PLA blend impeding the mobility of the PBAT segment. Similar findings were reported by Ding *et.al.,* [3] in their work on the PLA-PBAT-PLA tri-block copolymers. Meanwhile, the T_g PLA in the co-50PBAT/PLA blend remain unchanged with the evidence of its value at 59.9 °C.

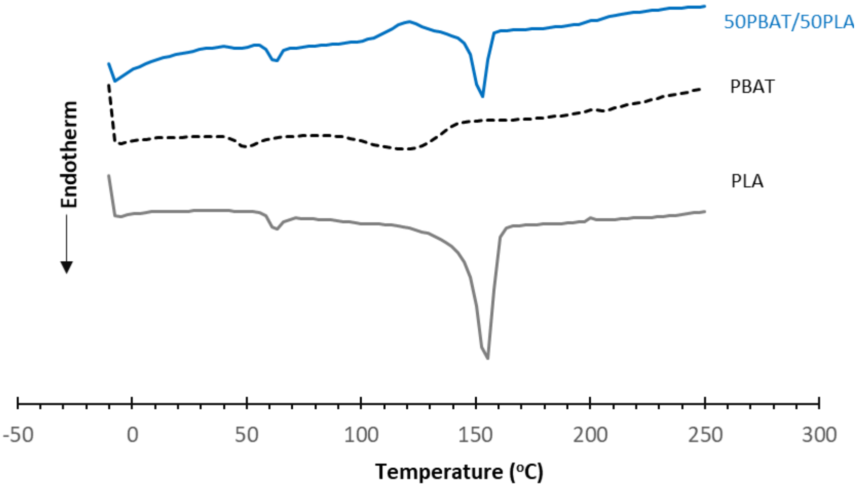


Fig. 3. DSC curves for PLA, PBAT and co-blend 50PBAT/PLA

For the PBAT and PLA melting temperatures, T_m the values are 121.3 °C and 154.3 °C, respectively. Interestingly, T_m PBAT demolished in the co-50PBAT/PLA blend and a significant crystallization peak referred as T_{cc} co-50PBAT/PLA blend is appeared at 145.3 °C. PLA has significantly hindered the crystallization of PBAT in the co-50PBAT/PLA blend. While the T_m PLA in the blend gradually shifted to lower T_m which at 152.1 °C, suggesting that PBAT has influenced the PLA crystallization in the co-50PBAT/PLA blend. The findings suggested that the PBAT chains in the co-50PBAT/PLA blend acted as infections, disrupting the crystallisation of the PLA chains and, as a result, lowering the T_m PLA in the co-50PBAT/PLA blend.

The effect of PS on the thermal properties co-50PBAT/PLA blend are further characterized and the thermograms are shown in Figure 4 and the data of T_g PBAT, T_g PLA, T_m PS, T_m PLA and T_{cc} of co-50PBAT/PLA blend are tabulated in Table 3. From Table 3, it appears that the addition of PS caused the T_g of PBAT and PLA in the co-50PBAT/PLA blends to progressively move toward one another, indicating that the PS may function as a compatibilizer for co-50PBAT/PLA blends.

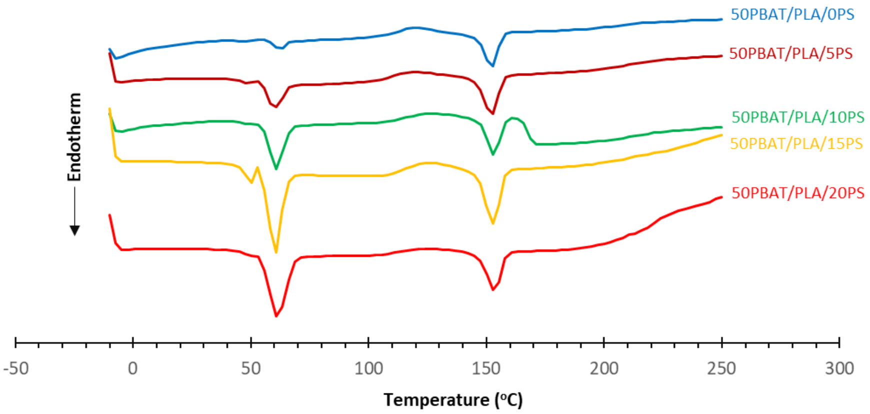


Fig. 4. DSC curves for co-50PBAT/PLA blend with 5, 10, 15 and 20 wt.% PS

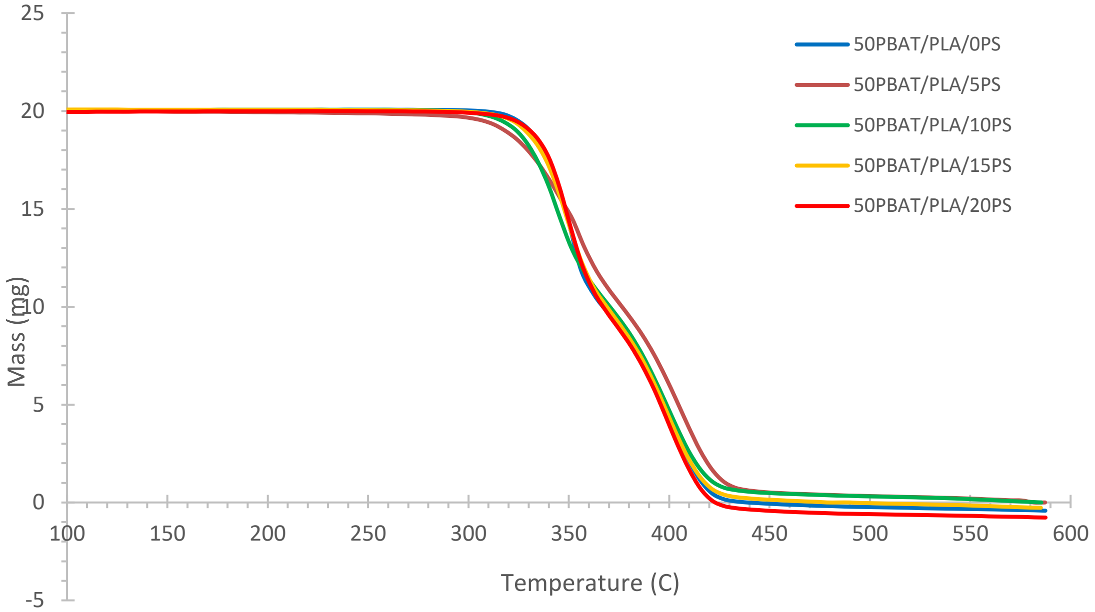
Table 3
Thermal properties of co-50PBAT/PLA blend with 5, 10, 15 and 20 wt.% PS

	T_g PBAT (°C)	T_g PLA (°C)	T_m PS (°C)	T_{cc} (°C)	T_m PBAT (°C)	T_m PLA (°C)
PBAT	44.9	-	-	-	121.3	-
PLA	-	60.1	-	-	-	154.3
PS	-	-	61.9	-	-	-
50PBAT/PLA/0PS	45.7	59.9	-	120.5	-	152.1
50PBAT/PLA/5PS	47.4	56.6	60.8	119.3	-	152.1
50PBAT/PLA/10PS	47.5	57.8	60.8	124.8	-	153.1
50PBAT/PLA/15PS	47.7	57.5	60.7	125.3	-	152.6
50PBAT/PLA/20PS	48.2	57.2	60.8	124.8	-	153.2

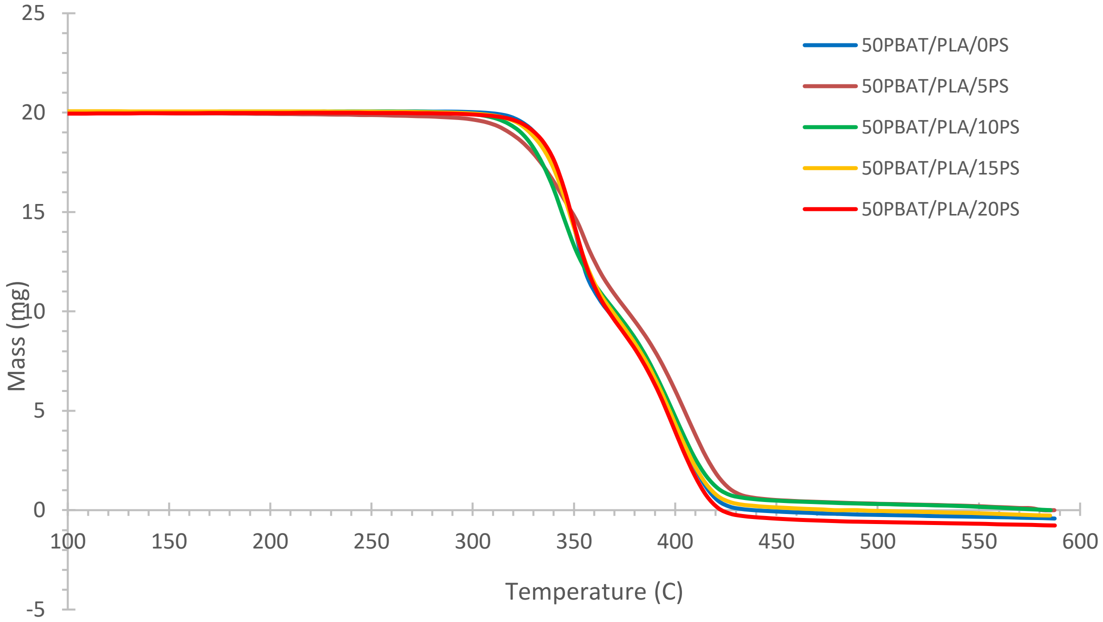
The thermal properties were investigated in more detail by also studying other parameters such the cold crystallization temperature (T_c) of the co-50PBAT/PLA blends without PS was 120.5 °C. Addition of PS has allowed the co-50PBAT/PLA blend to crystallize at the range of temperature between 119.3 °C to 125.3 °C before the melting of co-50PBAT/PLA blend occurs around 152.1 °C. The T_c of samples increased with the addition of PS, which indicated that PS hindered the crystallization in the co-50PBAT/PLA blends. Furthermore, the melting temperature of co-50PBAT/PLA blend which might be corresponds to melting PLA does not appreciably change when the PS is added.

3.4 Thermogravimetric Analysis (TGA)

Figure 5(a) depicts the thermogravimetric analysis whereas Figure 5(b) depicts the derivative thermogravimetric (DTG) of co-50PBAT/PLA blends with varying PS concentrations.



(a)



(b)

Fig. 5. (a) TG and (b) differential TG (DTG) curves of co-50PBAT/PLA at different PS addition

Table 4 sets out the thermogravimetric data for all compositions, including the temperature at 5 wt.% weight loss (T_{d5}) and defines the decomposition temperature ($T_{\max}$), where the temperature gave the maximum weight loss rate from TGA for PBAT, PLA, co-PBAT/PLA blends with varying PS addition.

PLA has smaller T_{d5} compared to the PBAT. As observed in Figure 5(a), by adding PS up to 20 wt.% to the co-50PBAT/PLA blend, the T_{d5} was not affected significantly and disrupted the thermal stability of the co-50PBAT/PLA blend, however, added PS has influenced the degradation temperature of $T_{\max}$ PLA.

The degradation of neat PBAT and PLA occurred in a single step, starting at 329.1 °C for PBAT and 295.1 °C for PLA through a final temperature of 455.3 °C and 393.0 °C with a DTG peak at 405.3 °C and 362.1 °C, respectively. In co-50PBAT/PLA blends, the $T_{\max}$ values of PBAT and PLA were measured between the PBAT and PLA values. In Figure 5(b), two-step decomposition of co-PBAT/PLA blends with varying PS addition was obviously noted. Evidently, addition of more than 5 wt.% PS to the co-50PBAT/PLA blend significantly increased the rate of degradation with the evidence shown by the co-50PBAT/PLA5PS blend DTG curve.

As shown in Table 4, the $T_{\max}$ PLA of co-50PBAT/PLA blend has decreased to more 17 °C with 10 wt.% PS. While, the $T_{\max}$ PBAT was insignificantly changed, suggesting that PS did not affect the thermal stability of PBAT in the co-50PBAT/PLA blend in comparison to PLA.

Table 4
Td_5 of co-50PBAT/PLA blends at different PS addition

Composition	T_{d5} (°C)	T_{maxPLA} (°C)	$T_{maxPBAT}$ (°C)
PBAT	369.5	-	405.3
PLA	329.5	362.1	-
50PBAT/PLA/0PS	329.3	350.4	403.7
50PBAT/PLA/5PS	318.3	346.2	398.1
50PBAT/PLA/10PS	323.7	345.4	397.9
50PBAT/PLA/15PS	329.2	350.8	397.8
50PBAT/PLA/20PS	329.4	350.1	398.0

3.5 Mechanical Properties

Table 5 exhibits the tensile properties of the 50PBAT/PLA with addition PS from 5 to 20 wt.%. The tensile strength and Young’s modulus decreased with increasing PS content in PBAT/PLA blends. This observation is agreed with the demolished of PBAT crystallization as shown in Figure 4 that may contribute to less strength to PBAT/PLA blend when PS was added to this blend. Furthermore, this result was also presumably due to the softening effect of PS. This result is consistent with Nagy *et al.,* [13] who claim that impact strength decreases with increasing starch content.

Table 5
Tensile properties

	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (MPa)
50PBAT/PLA/0PS	3.95	4.87	157
50PBAT/PLA/5PS	3.9	6.3	145
50PBAT/PLA10PS	3.66	7.27	154
50PBAT/PLA/15PS	2.82	9.52	126
50PBAT/PLA/20PS	3.34	8.14	144

Next, the mechanical properties (elongation at break) of the PBAT/PLA increased with PS content up to 15 wt.%. The elongation at break of PBAT/PLA/15PS was approximate 200% (almost 2 times that of the PBAT/PLA/0PS, demonstrating the good flexibility of PBAT/PLA blends. Evidently, this characteristic is due to the compatibilization of PS to PBAT/PLA blends as confirmed by the shifting of T_g PBAT and PLA in the Figure 4. Zhou *et al.,* [19] claimed that the elongation of specimen with addition of 0.4% novel phosphorus-based ionic liquid compatibilizers in PBAT/PLA was almost 4 times of the pristine PBAT/PLA composite demonstrated the good flexibility and tenacity of the specimen. The addition of 20 wt.% of PS to the 50PBAT/PLA formulation revealed a reduction in elongation at breaks that was considered the optimum PS content in the 50PBAT/PLA formulation. However, the higher elongation at break for specimen 50PBAT/PLA/15PS resulted in low tensile strength and

Youngs's modulus. According to El-hadi *et al.,* [4], the addition of plasticizer to PLLA composite increases flexibility and elongation at break by greater than 200%.

4. Conclusion

In this presence work, PS is established as an alternative compatibilizer for PBAT/50PLA blend system based on the rheology, thermal and mechanical properties. From the rheological analysis, the addition of PS in PBAT/PLA blend shows good flowability at temperature 175 °C, exhibits pseudoplastic behaviour thus making it suitable as a feedstock for injection moulding application. At high temperatures, the thermal stability of the PBAT/PLA blend remained unchangeable with the PS addition. From the mechanical properties point of view, addition of PS up to 15 wt.% has reduced the tensile strength and Young's modulus, but improve the elongation at break approximately 200%, demonstrated the good flexibility of composites due to the compatibilization PBAT/PLA blend with the added PS.

Acknowledgement

Financial support from the Ministry of Higher Education Malaysia under the Fundamental Research Grant Scheme (FRGS/1/2019/TK02/UITM/02/19) and Circular+ Industrial Research Laboratory, College of Engineering, Universiti Teknologi MARA (UiTM).

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