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The Effect of Poly(butylene adipate-co-terephthalate) (PBAT)/ Oil Palm Frond (OPF) Composite with Palm Stearin as Compatibiliser

Istikamah Subuki[1*], Muhammad Danish Zuhairi Azrin[1], Ameer Azali Mohamed Ali[1], Suhaiza Hanim Hanipah[1], Nur Azrini Ramlee[1]

[1]*School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

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ABSTRACT

The chemical composition of palm stearin (PS), with its highly saturated fats and triglycerides, makes it a potential candidate to serve as a compatibilizer. This study investigated the effect of PS as a compatibiliser on the thermal and mechanical properties of poly(butylene adipate-co-terephthalate) (PBAT)/oil palm frond (OPF) composites. In this paper, a biodegradable composite was prepared by blending the PBAT with OPF as a filler, where it was subsequently mixed with PS as a compatibiliser. A mixture of PBAT, OPF, and PS was melted using an internal mixer at 160 °C and 60 rpm for 12 minutes. The various formulations were then subjected to torque evaluation and thermal and mechanical analysis to characterise the samples. The torque analysis indicated that the PBAT was compatible with 30 wt.% OPF. The chemical bond between PS, PBAT, and OPF is responsible for the decrease in the melting point and degradation temperature of the PBAT/OPF composite with increasing PS concentration. The presence of PS in the PBAT-OPF composite improved its flowability and moulding process. Accordingly, the tensile strength and ductility showed improvement with the addition of 7.5% PS in the PBAT70/OPF30 composite. Meanwhile, the addition of PS resulted in a decrease in Young's modulus. The results indicated the suitability of the PS as a compatibiliser for the PBAT/OPF composite.

INTRODUCTION

The growing concern over plastic pollution and its adverse environmental effects has led to an increased focus on developing sustainable alternatives to fossil-based polymers. Poly(butylene adipate-co-terephthalate) (PBAT) has emerged as a promising biodegradable polymer due to its ability to decompose when buried in soil completely (Jian et al., 2020). Despite being composed of non-renewable resources, PBAT exhibits superior biodegradability compared with conventional plastics, making it a suitable candidate for anti-plastic pollution strategies (Tadele et al., 2023). Oftentimes, natural polymers, such as starch, cellulose, and protein, are blended as fillers to lower the manufacturing expense of PBAT - which

[1*] Corresponding author. *E-mail address*: istikamah@uitm.edu.my
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is one of the main barriers to its use - while preserving its biodegradability (Kim et al., 2023). However, PBAT faces challenges in terms of limited thermal stability, as well as poor interfacial adhesion and poor mechanical properties, all of which restrict its application in high-performance fields (Mochizuki & Hirami, 1997).

To enhance the mechanical properties and expand the application potential of PBAT, researchers have explored the incorporation of natural reinforcements, such as leaves and plant fibres, as reinforcing agents in PBAT-based biodegradable matrices (Elkhaoulani et al., 2013; Scaffaro et al., 2022). These efforts aim to reduce costs and improve the overall resilience of bioplastics. Notably, previous studies focusing on PBAT blended with leaves and fibres from the Chamaerops humilis dwarf palm have successfully increased the mechanical properties of resulting biocomposites. The improvements in mechanical performance were attributed to the development of a robust interphasic area between the filler and the polymer matrix, which is achieved through suitable modifications to the Halpin-Tsai model that considers the crucial role of the interphase (Scaffaro et al., 2022).

However, blending PBAT with organic fillers can lead to increased stiffness at the expense of reduced toughness, thereby limiting the potential application fields of such biodegradable composites. One approach to address this challenge is the use of compatibilisers, which are substances that promote compatibility between the hydrophilic nature of natural fillers and the hydrophobic PBAT matrix (Pinheiro et al., 2014). Compatibilisers play a vital role in enhancing the interfacial adhesion and mechanical properties of the resulting composites (Fink, 2013; Huang et al., 2021). Among the potential compatibilisers, palm stearin (PS) has garnered attention due to its abundant availability, cost-effectiveness, and renewable nature.

Palm stearin is a by-product obtained during the processing of palm oil and is characterised by its waxy nature, high concentration of saturated fats, and triglycerides (Pande et al., 2012). It was found that it can act as a plasticiser in natural rubber compounds, where it improves processability and enhances mechanical properties (Barlow, 1993; Surya et al., 2013). Moreover, the chemical composition of palm stearin, with its highly saturated fats and triglycerides, makes it a potential candidate to serve as a compatibiliser in PBAT/OPF composites (Pande et al., 2012). Therefore, this paper aims to evaluate the effect of PS as a compatibiliser in PBAT/OPF composites through melt blending, thermal analysis, and mechanical properties.

METHODOLOGY

Materials

This work used a commercial grade of PBAT resin supplied by Shanghai Hengsi New Material Science and Technology Co. Ltd., and refined, bleached, deodorised (RBD) palm stearin (PS) purchased from the Lipidchem Sdn. Bhd. The OPF was supplied from PPK Kuala Langat and was cut into small pieces, then dried in the oven at a temperature of 105 °C for 24 hours and sieved at 100 µm.

Preparation of the polymer composite

Initially, composites were prepared using PBAT/OPF ranging from 25 wt.% to 35 wt.%, as shown in Table 1. Then, the PS, ranging from 2.5 wt.% to 7.5 wt.% (Adriana et al., 2018), was added to the 70 wt.% PBAT/30 wt.% OPF. An internal mixer (HAAKE PolyLab OS RheoDrive7, Thermo Scientific) was used, and the mixing temperature and speed of the mixer screw were set at 160 °C and 60 rpm, respectively, for 12 minutes. Table 1 shows the composition of the PBAT/OPF composites, as well as the added PS concentration.

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Table 1. Composition of the PBAT/OPF/PS composites

Composite formulation	PBAT (wt.%)	Oil palm frond (wt.%)	PS
PBAT75/OPF25	75	25	-
PBAT70/OPF30	70	30	-
PBAT65/OPF35	65	35	-
PBAT/OPF/PS2.5	70	30	2.5
PBAT/OPF/PS5.0	70	30	5
PBAT/OPF/PS7.5	70	30	7.5

The homogeneity of the composite was estimated using torque rheometry by monitoring the torque variation. This is the preliminary study to investigate the homogeneity of the composite blend formulation. The measuring principle of this technique is based on the resistance that the material opposes to the rotation of the blades. Afterward, the composite resin was crushed using a compact crusher (HMRV50-19, Rexmac) to form a palleted size.

Thermal characterisation

The thermal analysis on the PBAT/OPF/PS composite was accomplished using a thermal gravimetric analyser (TGA; SDTA581e, Mettler Toledo) with a sample of approximately 10 mg that heated at a heating rate of 10 °C/min from 30 °C to 750 °C under a 50 mL/min nitrogen atmosphere. Differential scanning calorimetry (DSC; Stare system, Mettler Toledo) was then used to identify the melting temperature (Tm) of the sample. In this analysis, 5 mg of the samples were heated at a heating rate of 10 °C/min at a temperature ranging from 40 °C to 250 °C.

Compaction process

The tensile specimen was prepared using the hot pressing and compaction method. The PBAT/OPF/PS composite granules were shaped into dumbbell-shaped specimens by a hot press machine (QC-602A, Cometech) at a temperature ranging from 170 °C to 200 °C for preheating and compression. The procedures allowed the granules to melt and spread out between the plates. Subsequently, the melted PBAT/OPF/PS composites were cooled at 50 °C under a pressure of 5 MPa for 10 min. A desiccator was used to keep the samples free of moisture prior to their analysis.

Mechanical properties

The mechanical properties of the specimen (tensile strength, elongation at break, and Young's modulus) of the PBAT/OPF/PS composites (5 samples) were measured in accordance with the ASTM D882-10 test method using a universal testing machine (H50KT, Instron 3382, Titinus Olsen). The machine was equipped with a 2-kN load cell with ±0.5% accuracy and a crosshead speed set at 25 mm/min. At least five specimens were used for each test.

RESULTS AND DISCUSSION

Effect on mixing characteristic

Fig 1 displays the torque evolution curves for the PBAT/OPF composites and PBAT/OPF/PS formulated at different blending ratios. It displays the relationship of torque variations with the mixing time. The amount of work energy required for the dispersion and distribution of PBAT, OPF powder, and PS powder is indicated by the mixing torque, which is proportional to the mixer's shear stress. Typically, the torque profile elevates with mixing time until a maximum peak is reached.

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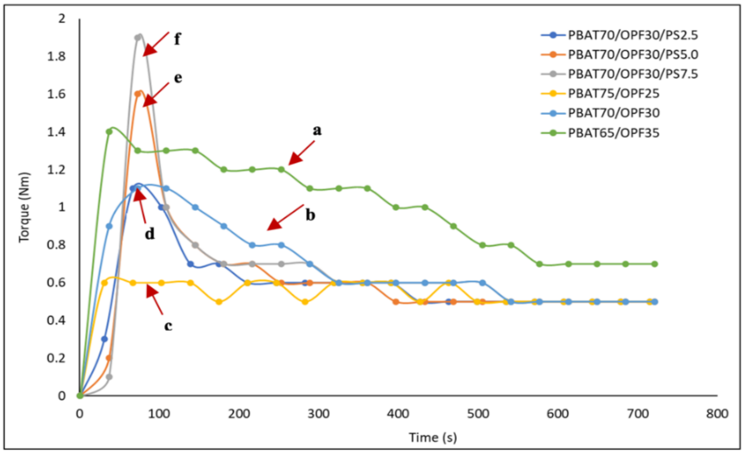

Fig. 1. Torque evolution curves of (a) PBAT65/OPF35, (b) PBAT70/OPF30, (c) PBAT75/OPF25, (d) PBAT/OPF/PS2.5, (e) PBAT/OPF/PS5.0, and (f) PBAT/OPF/PS7.5.Nm

The torque profile clearly shows a decrease in the torque value from 1.4 Nm to 0.6 Nm when the PBAT increased from 65 wt.% to 75 wt.%. This is attributed to the higher concentration of the lower molecular weight PBAT (110 °C) compared with OPF (1730 °C), which expands at higher temperatures, leading to lower viscosity and reduced torque value. For the PBAT/OPF composite containing 25 wt.% OPF, the torque was lower with the instability phenomenon. For the composite PBAT70/OPF30, the torque stabilised at a steady level in a short time of 300 seconds, indicating a uniform mixing. In contrast, PBAT65/OPF35 requires more time to stabilise the torque value. This suggests that a longer mixing time of 600 seconds is necessary to achieve a homogenous mixing. Based on the curves in Fig 1, PBAT70/OPF30 is considered a suitable composition of composite for this study.

The addition of PS as a compatibiliser results in significant changes, where the PBAT/OPF/PS2.5 composite displays a substantial reduction in maximum torque values at 1.1 Nm. This indicates enhanced compatibility and dispersion facilitated by the lubricating and compatibilising effects at 2.5 wt.% PS. This trend continues in the composition PBAT/OPF/PS5.0, with a maximum torque at 1.6 Nm that rapidly decreased to consistently low torque values, thus suggesting an optimal range where 5 wt.% PS effectively promotes efficient mixing and compatibility. However, it was observed that while the composition of PBAT/OPF/PS7.5 at 1.9 Nm was at its peak, it still demonstrated low torque values and no substantial improvement over the 5 wt.% PS composition. This may indicate a plateau effect, suggesting that beyond a certain PS content, further enhancements in compatibility and dispersion are limited. A similar trend was documented by previous studies (Sudari et al., 2017), where the addition of a surfactant to a bio composite required higher rotational forces for achieving proper surfactant dispersion, leading to an analogous observation.

Effect on thermal properties

Using the DSC method, thermal characteristics are assessed by heating a sample and monitoring the flow of heat. The study of the thermal properties of the PBAT/OPF composite can be done by plotting the heat flow (in mW) versus the sample temperature (in °C). Table 2 shows the melting temperature (*Tm*) of PBAT and PS, and the crystalline ratio (*Xc*) for the three various PS concentrations of the PBAT/OPF/PS composites corresponding to the PBAT/OPF composite and PS. The values were from the corresponding DSC curves displayed in Fig 2.

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Table 2. Thermal properties of the PBAT/OPF/PS composite blends

PBAT/OPF/PS	PBAT			PS		
	T_m (°C)	ΔH_m (J/g)	X_c	T_m (°C)	ΔH_m, (J/g)	X_c
PBAT70/PF30	122.64	4.86	6.10	-	-	-
PBAT/OPF/PS2.5	121.23	8.86	11.10	47.50	1.88	35.41
PBAT/OPF/PS5.0	121.71	6.83	8.56	47.55	3.02	47.04
PBAT/OPF/PS7.0	121.9	6.14	7.69	47.60	3.41	58.57

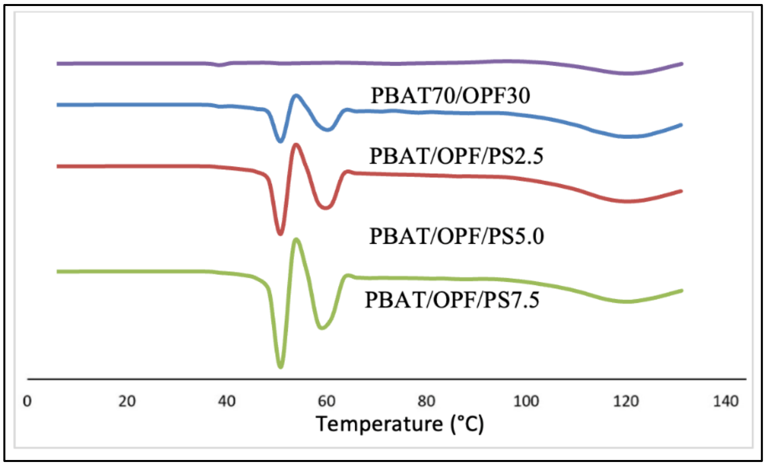

Fig. 2. DSC curves obtained for PBAT/OPF/PS composite blends.

From Fig 2, The PBAT/OPF composite curve shows a relatively minimal peak, indicating the presence of an amorphous phase. Since there was no evidence of a crystallisation peak observed during the heating cycle, the PBAT/OPF combination is regarded as amorphous. For the PBAT/OPF/PS composites, the *Tm* was found to have decreased with increased composition of PS. The PBAT/OPF/PS composites DSC curves revealed crystallisation, which may have been caused by the inclusion of PS. From the calculated *Xc*, the value of crystallinity of the composites increased with the addition of PS as shown in Table 2. The kinetics of the polymer's crystallisation were enhanced by the addition of GMS compatibiliser added to pure poly-L-lactic acid (PLLA) (Alamri et al., 2020).

Fig 3 shows the TGA curves for the PBAT/OPF and PBAT/OPF/PS composites. The temperature at 5 wt.% weight loss (T_d5) and maximum decomposition temperature (T_max) were determined from the corresponding DTG curves. According to the data shown in Table 3, the thermal stability of the PBAT/OPF composite was higher than that of the PBAT/OPF composites with PS added. The 5 wt.% loss (T_d5) point of the PBAT/OPF/PS composites shifted to a lower temperature compared to the PBAT/OPF composites. The addition of 2 wt.% to 7.5 wt.% PS to the PBAT/OPF composites caused no significant reduction in the T_d5 value. Previous studies showed that the addition of compatibilisers in PLA/PP resulted in no relevant changes in thermal stability (Gonçalves et al., 2020).

The temperature at which the maximum rate of weight loss occurs is known as the decomposition temperature (Ng et al., 2018; Tsou et al., 2022). Compared with the PBAT/OPF, the thermal degradation (T_max) of the PBAT/OPF/PS composites shifted slightly toward lower temperatures. The T_max value was reduced from 463.04 °C for the PBAT/OPF to 462.60 °C when 2.5 wt.% PS was added. The addition of 5.0 wt.% and 7.5 wt.% PS to the PBAT/OPF composite further lowered the T_max to 456.25 °C (PBAT/OPF/PS5.0) and 448.41 °C (PBAT/OPF/PS7.5), respectively. It was clear that the presence of the PS compatibiliser had facilitated the thermal degradation of the polymer composite. This is in agreement

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with the work of other researchers on the decomposition of the poly[(R)-3-hydroxybutyric acid] (PHB) (Scaffaro et al., 2022). It was found that the value of T_max had dropped from 415 °C to approximately 320 °C with the addition of GMS/triacetin (TA) into the polymer. However, the introduction of polypropylene-grafted-maleic anhydrides (MAgPP) as a compatibiliser slightly increased the T_max of the composite (Eszer & Ishak, 2018). Table 2 also shows that the decrement in weight losses demonstrated a slight improvement in thermal stability from the addition of the PS compatibiliser. This is attributed to the chemical bond between PS with PBAT and OPF, which may have enhanced the interfacial adhesion.

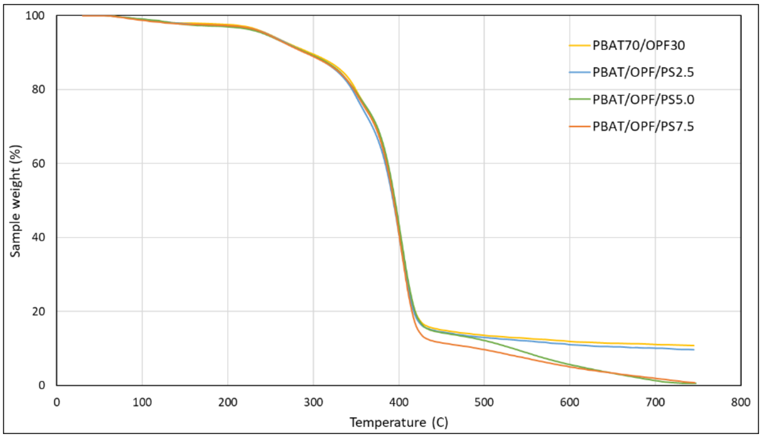

Fig. 3. TGA curves of the PBAT/OPF/PS composites.

Table 3. Thermogravimetric data for PBAT/OPF/PS composites

Formulation	T_{d5} (°C)	T_{max} (°C)	Residue (wt.%) (at 700 °C)
PBAT70/OPF30	250.53	463.04	11.08
PBAT/OPF/PS2.5	243.56	462.60	10.03
PBAT/OPF/PS5.0	243.34	456.25	3.3
PBAT/OPF/PS7.5	243.55	448.41	1.21

Effect of PS addition on mechanical properties

Mechanical testing provides a good indication of the composite physical properties. Fig 4(a) shows the tensile strength and elongation, and Fig 4(b) shows Young's modulus of the PBAT/OPF composite with different PS content. The presence of compatibiliser significantly improves the dispersion of composites, provides better homogeneity, and increases tensile strength (Mantia et al., 2017; Zoukrami et al., 2008). Fig 4(a) portrays that the tensile strength of PBAT/OPF has improved slightly with the addition of PS, suggesting that the added PS is compatible with the PBAT/OPF composite. The presence of PS in the PBAT and OPF composites may enhance adhesion, contributing to the slight increase in tensile strength. The addition of 7.5 wt.% PS significantly improved the level of ductility.

From Fig 4(a), it was observed that the elongation at break increased with the addition of PS content, which implied that the toughness of the PBAT/OPF/PS composites had improved significantly. The addition of the compatibiliser up to 3 wt.% in thermoplastic formulations had little impact on the tensile characteristics (Liu et al., 2001). In another relevant work, it was found that adding PHB and GMS/TA as plasticisers to the PLLA blends improved their elongation at break when compared to pure PLLA (Alamri et al., 2020).

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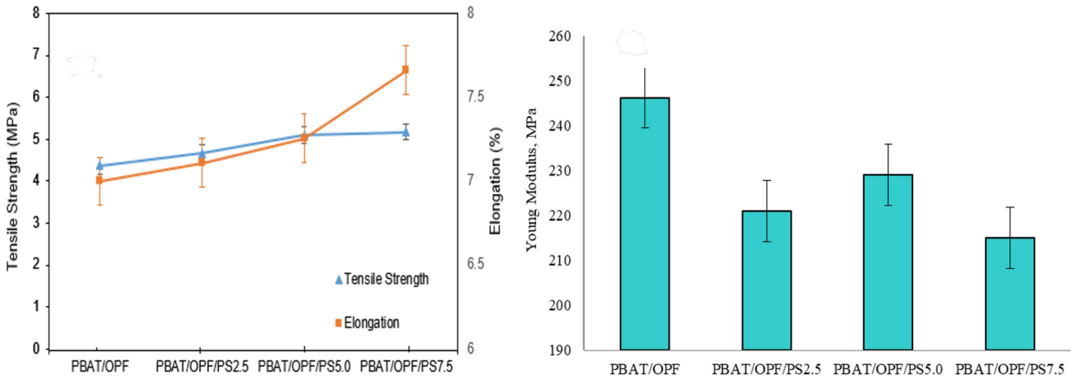

Fig. 4. (a) TGA of the tensile strength and elongation and (b) Young's modulus of PBAT/OPF and PBAT/OPF/PS composites.

Meanwhile, Young's modulus (Fig 4(b)) decreased by 25 MPa, from 246 MPa to 221 MPa, with the addition of 2.5 wt.% PS. The decreasing modulus may be due to the softening effects on the entire blend, resulting in a slight reduction in stiffness (Hedayati et al., 2020). The softening effects continue further with PS addition. The highest Young's modulus value for PBAT/OPF/PS composites was recorded at 5 wt.% with PS addition, which was 230 MPa. A polymer with a high Young's modulus and tensile strength demonstrated a lower elongation at break value (Amjadi & Fatemi, 2020; Muslim et al., 2023). PBAT/OPF/PS5.0 exhibited an elongation at a break of 5.1% at the time the maximum value of Young's modulus was observed.

Based on the data presented in Fig 4, the tensile strength of the PBAT/OPF/PS composites reaches its maximum value at 7.5 wt.% PS content. Evidently, the PS had contributed to improving the brittleness of the PBAT/OPF composites. When the PS content was 7.5 wt.%, the PBAT/OPF composites maintained high tensile strength and excellent elongation upon breaking, as well as better mechanical characteristics.

CONCLUSION

The study strategically explored the incorporation of PS as a compatibiliser to enhance the tensile strength, elongation, and thermal stability of PBAT/OPF composites. The investigation successfully demonstrated that the addition of PS enhanced the degree of compatibility between PBAT and OPF, leading to remarkable advancements in thermal and mechanical properties. In terms of mechanical properties, adding up to 7.5 wt.% PS has improved the level of ductility and tensile strength, even though the Young's modulus is the lowest compared to others. This is likely due to the softening effects produced by the good adhesion of such a composite blend.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits, commercial or financial conflicts and declare the absence of conflicting interests with the funders.

AUTHORS' CONTRIBUTIONS

The authors confirm their contribution to the paper as follows: **study conception and design**: Muhammad Danish Zuhairi Azrin, Istikamah Subuki, Ameer Azali Mohamed Ali, Suhaiza Hanim Hanipah, Nur Azrini Ramlee; **data collection**: Muhammad Danish Zuhairi Azrin, Istikamah Subuki, Ameer Azali Mohamed Ali, Suhaiza Hanim Hanipah, Nur Azrini Ramlee; **analysis and interpretation of results**: Muhammad Danish Zuhairi Azrin, Istikamah Subuki, Ameer Azali Mohamed Ali, Suhaiza Hanim Hanipah, Nur Azrini Ramlee; **draft manuscript preparation**: Muhammad Danish Zuhairi Azrin, Istikamah Subuki, Ameer Azali Mohamed Ali, Suhaiza Hanim Hanipah, Nur Azrini Ramlee. All authors reviewed the results and approved the final version of the manuscript.

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