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Effect of Palm Stearin as a Compatibilizers in PBAT/Zein Blends Biodegradable Polymer

Journal of Electrical Systems

Abstract: - In this study, the introduction of PBAT was conducted through reactive blending with Zein, a renewable natural biopolymer derived from corn, in the presence of Palm Stearin (PS); one of the components in palm oil. The objective of this study is to examine the impact of mechanical forces on PBAT/Zein composites, both with and without the presence of PS. The PBAT/Zein and PBAT/Zein/PS blends will be subjected to various experiments, including mechanical testing and thermal analysis. Variation of different formulations of composition, comprised of PBAT, Zein and PS were used in this study. The thermal and mechanical properties of the PBAT/Zn blend were assessed through the utilization of mixing characteristics as well as thermal and mechanical properties. Based on the findings, it has been determined that the optimal composition for the PBAT/Zein blend is achieved when the blend contains 10% zein content. Furthermore, the optimal composition of PBAT/Zein/PS is achieved when 10 phr of PS are included in the blend.

Keywords: Palm Stearin, PBAT, Zein, Compatibilizer, Mechanical properties

1.0 Introduction

PBAT (polybutylene adipate terephthalate) is an aliphatic-aromatic co-polyester that is biodegradable (Guo et al., 2015). PBAT can be manufactured via the polycondensation reaction of butanediol (BDO), adipic acid (AA), and terephthalic acid (PTA) (Jian et al., 2020). The aromatic monomers have superior thermal stability and mechanical qualities, whereas the aliphatic monomers are flexible and biodegradable (Wei et al., 2019). PBAT's limited mechanical qualities and excessive cost have prevented its widespread adoption (Wei et al., 2019). It has been reported that blending PBAT with natural polymers including soy protein isolate (SPI) (Guo et al., 2015), soy meal (Zhou et al., 2013), cassava starch (Brandelero et al., 2010), corn gluten meal (Reddy et al., 2014), and pine resin derivatives such gum rosin (GR) and pentaerythritol ester of GR (UT) (Pavon et al., 2020), could balance the cost and characteristics. In the previous study, Wei et al. (2019) achieved the development of a new biodegradable polymer by blending PBAT and Zein and adding PEDGE as a compatibilizer.

45–50% of the protein in corn is composed of zein, the primary storage protein (Shukla & Cheryan, 2001). Corn, one of the highest-yielding crops in the world, is an essential food crop used to produce corn starch (Wei et al., 2019). As a result, the scraps were frequently marketed as a low-quality source of protein for animals or even dumped into the river, resulting in severe environmental degradation (Wei et al., 2019). Exploring more effective uses for zein, a renewable natural biopolymer, would therefore be crucial for supporting corn sector development and environmental protection, with major economic and social benefits (Wei et al., 2019). In addition, Zein Plastics could absorb atmospheric moisture, which could drastically lower its tensile strength and modulus (Tian et al., 2009). This is supported by (Wei et al., 2019) allegation that pure zein is not thermoplastic and has shallow mechanical characteristics.

The addition of other natural polymers resulted in decreasing the tensile strength (MPa) and elongation at break (%) significantly. It may be owing to the poor compatibility and interfacial adhesion between PBAT and natural polymers, which reduces mechanical characteristics. Thus, in past literature, modified natural polymers, such as soy meal (Zhou et al., 2013), and corn gluten meal (Reddy et al., 2014), was added to PBAT to improve its mechanical characteristics. The research conducted by (Zhou et al., 2013) demonstrated a significant decrease

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in the tensile strength and elongation of PBAT/SM compared to neat PBAT. However, it was observed that the modulus of the blends increased. The inadequate interfacial adhesion observed between PBAT and SM results in a decrease in mechanical properties. (Wei et al., 2019) found that the addition of PEDGE to PBAT/Zein as a compatibilizer improved the material's mechanical characteristics. The reaction of the epoxy group with PBAT and zein will enhance the interfacial compatibility between PBAT and zein, which is more advantageous for enhancing the mechanical properties of the PBAT/zein mix and the stability of zein in the PBAT matrix (Wei et al., 2019). The results show that PBAT/Zein/PEGDGE blends with a compound of 90/10/5 have the optimum results among the other compound in their study.

Palm oil is separated into two principal components, a solid fraction known as palm stearin (PS) and a liquid fraction known as palm olein (PO) (Subroto & Nurannisa, 2021). Palm stearin is the harder fraction of palm oil, having a larger concentration of saturated fatty acids and triacylglycerol (TAG) with a melting point of 48–50°C (Pande et al., 2012). Palmitic acid ranges from 49 to 68%, whereas oleic acid ranges from 24 to 33% (Pande et al., 2012). The composition, physical characteristics, solid fat content, and iodine values of palm stearin vary widely (Pande et al., 2012). PS is solid at normal temperatures due to its high concentration of saturated fatty acids (Ramadan, 2019; Sofwan Sinaga & Siahaan, 2018; Subroto & Nurannisa, 2021). It comprises around 47-74% palmitic acid, 16-37% oleic acid, 4-6% stearic acid, 3-10% linoleic acid, and 1-2% myristic acid (Rohmah et al., 2019). Thus, the aim of this study is to investigate the effect of palm stearin as a compatibilizer in PBAT/Zein blends. As well as, to investigate the effect of mechanical properties of PBAT/Zein with or without PS.

In this study, zein was employed as a filler to fabricate a novel material with favourable mechanical properties through reactive blending with poly (butylene adipate-terephthalate) (PBAT). The manufacturing process of pure zein into a specific material is challenging due to its non-thermoplastic nature and inadequate mechanical properties (Tian et al., 2009; Wei et al., 2019). Thus, palm stearin is added to the PBAT/Zein as compatibilizer to further increase the mechanical properties. The compatibilizer serves to decrease the interfacial energy, thus enhancing the adhesion between the polymer phases. This will lead to the efficient distribution of components and minimize agglomeration (Jayakumar et al., 2022).

2.0 Methodology

2.1 Drying

Palm Stearin, Zein (Sigma-Aldrich (USA) and PBAT grade 801T were dried in a vacuum oven at 40 °C for 24 h before blending, to remove moisture for avoiding hydrolytic degradation.

2.2 Compounding process

The PBAT polymer matrix will be blended with varied proportions of Zein and PS filler particles, as illustrated in **Table 1**. At room temperature, zein and PBAT were combined using a mixer before being stirred for 15 minutes at 180 ℃ in an intensive mixer (Haake Rheomix OS Thermo Scientific). PS was added to the intensive mixer with the zein and PBAT mixture to add to the PBAT/Zn blends.

Table 1: Compositions of PBAT/Zein Blends

Composition (wt%)			Phr
Sample	PBAT	Zein	PS
95PBAT/5Zn	95	5	-
90PBAT/10Zn	90	10	-
85PBAT/15Zn	85	15	-
90PBAT/10Zn/5PS	90	10	5
90PBAT/10Zn/10PS	90	10	10
90PBAT/10Zn/15PS	90	10	15

The obtained PBAT/Zn blends with and without PS were labelled PBAT/Zn: 90/10 (mass ratio) and for PBAT/Zn/PS: 90/10/5, respectively. The obtained sample was compression-molded on a vulcanizing machine (GT-7014-H30C, Taiwan High Speed Rail Testing Instrument Co., Ltd. Taiwan, China) for 10 minutes at 180 ℃ and 30 MPa molding pressure to create a dumbbell sheet (Wei et al., 2019).

2.3 Characterizations

A thermogravimetric analyzer, TGA (TGA: SDTA581e, Mettler Toledo) was utilized to examine the thermal stability of the blends. Under an environment of nitrogen, samples (7 - 8 mg) were heated from 50 to 600 °C at a rate of 10 °C/min (Wei et al., 2019). The purpose of this study is to determine the initial decomposition temperature (T_i) and maximum decomposition temperature (T_{max}) of PBAT/Zein, and PBAT/Zein/PS blends. All TGA will be performed three times. The melting temperature (T_m), and crystallization temperature (T_c) of PBAT/Zein, and PBAT/Zein/PS blends were examined using DSC (DSC: Star system, Mettler Toledo). To remove the thermal history of the sample, it was first heated to 150 °C at 50 °C/min and held for 6 minutes, then cooled to -80 °C at 10 °C/min and remained for 10 minutes, and finally reheated to 150 °C at a rate of 10 °C/min (Wei et al., 2019). In accordance with the Standard Test Method for Tensile Properties of Plastics (ASTM D638-14), dumbbell samples for tensile testing were cut from blend sheets with a cutter and conditioned for 48 hours at a relative humidity of 55% prior to testing. Using a Universal Tensile Machine Tinius Olson (Model:H50 KTA) with a crosshead speed of 20 mm/min (Wei et al., 2019), tensile tests were conducted. For each sample, at least 3 samples were evaluated to determine the mean value.

3.0 Results and discussion

3.1 Effects of Mixing Characteristics

Figure 1 illustrates the impact of incorporating Zein (Zn) into the PBAT/Zn blend on the mixing characteristics. The data clearly indicates that altering the amount of Zn addition leads to fluctuations in torque stability. Specifically, the torque value decreases up to a certain threshold of addition. In comparison to pure PBAT, as reported by (Aliotta et al., 2020) with a torque value of 0.82 Nm, it is evident that the addition of 5 to 15% of Zn into the blend results in a noticeable difference in torque values. Specifically, the torque value increases as the Zn content increases. According to a study conducted by (Chuakhao et al., n.d.), it was found that the torque value of pure PBAT increases when combined with Poly (lactic acid) (PLA) and Poly (butylene succinate) (PBS), yielding similar results.

The relationship between mixing torque and shear stress of the mixer suggests the amount of work energy required to disperse and distribute the PBAT/Zn blend (Subuki et al., 2012). The torque profiles demonstrate a significant reduction in the peak torque value, specifically from 1.3 Nm to 1.2 Nm, as the Zein content gradually increases from 5% to 10%. The observed phenomenon can be attributed to the higher concentration of Zein with a decreased molecular weight. The Zein variant in question demonstrates thermal expansion, leading to a decrease in viscosity and subsequently reducing the torque value. The torque of the PBAT/Zn composite, comprising 15wt.% Zein, demonstrated an increase to 1.3 Nm. The observed phenomenon can be attributed to the heightened friction caused by the elevated concentration of Zn. As a result, there was an increase in resistance on the rotor blades. The observed trend suggests that an elevated concentration of zein in the mixture has an impact. Based on the data depicted in **Error! Reference source not found.**, it can be inferred t hat the most favourable concentration of zein for the PBAT/Zn composite is 10wt.%.

Effect of addition of palm stearin in PBAT/Zein/PS blend

Fig. 2 illustrates the impact of incorporating palm stearin (PS) into the PBAT/Zn/PS blend on the mixing characteristics. The data clearly demonstrates that altering the amount of PS addition leads to variations in torque stability. Specifically, there is a decrease in torque value as the PS content increases. In comparison to the 90PBAT/10Zn mixture with a torque value of 1.2 Nm, as mentioned in section **3.1.1**, when incorporating 5 to 15 phr of PS into the blend, a noticeable disparity in torque values becomes apparent. Specifically, the torque value decreases by nearly half compared to the 90PBAT/10Zn blend as the PS content increases. The findings of (Jehsoh et al., 2021) support the observation that the torque value of the blend of Natural rubber (NR) and halloysite nanotubes (HNTs) increases with the addition of Modified PS.

Following the torque results obtained from the composite sample prepared without Palm Stearin (PS), additional investigations were conducted to assess the influence of PS addition (at concentrations of 5, 10, and 15 phr) as a compatibilizer on the composite comprising 10% Zein and 90% PBAT. The compositions are designated as 90PBAT/10Zn/5PS, 90PBAT/10Zn/10PS, and 90PBAT/10Zn/15PS, as depicted in **Fig. 2**. An observed reduction in the maximum torque value was noted when 5 phr of palm stearin (PS) were added to the blend, resulting in a decrease from 1.2 Nm to 0.7 Nm. A decreased torque value suggests improved compound processability. The use of PS has clearly improved the processability of the compounds. According to (Jehsoh et al., 2021), palm stearin (PS) exhibits a waxy texture, which allows it to serve as an internal plasticizer. This particular attribute contributes to a decrease in viscosity and improves the processability of diverse compounds. As the concentration of PS content in the blend increases, there is a corresponding decrease in the torque value. Based on our research, it has been observed that the inclusion of PS in the PBAT and Zein blends has resulted in a notable enhancement in their compatibility.

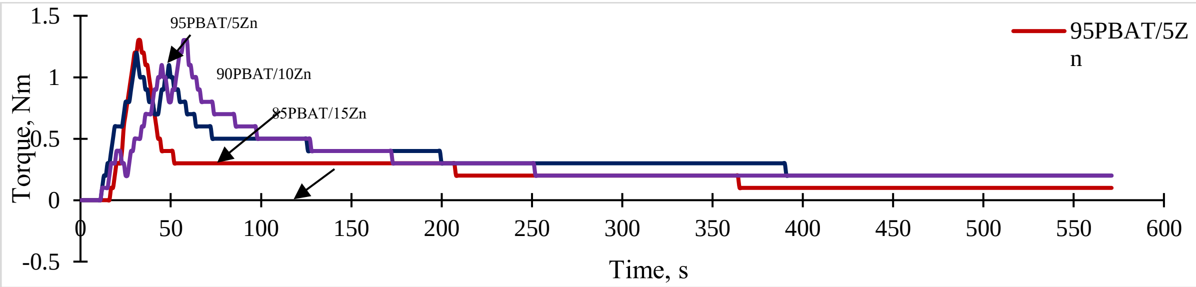

Fig. 1: Torque evolution curves for different composite compositions without PS (PBAT/Zn)

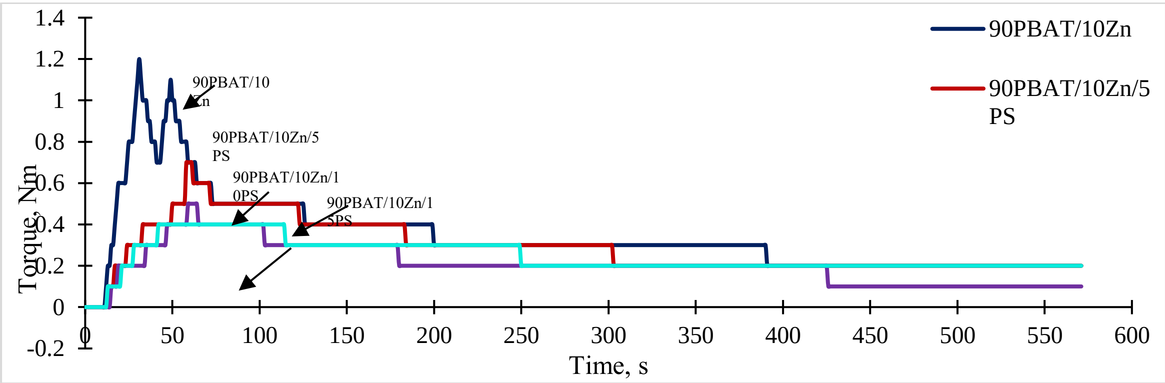

Fig. 2: Torque evolution curves for different composite compositions with PS (PBAT/Zn/PS)

3.2 Effects of Thermal Properties

Differential scanning calorimetry (DSC) is a scientific technique employed to ascertain thermal properties of a sample by subjecting it to controlled heating and precisely measuring the resulting heat flow. The thermal properties of DSC can be analysed by graphing the heat flow (mW) against the sample temperature (°C). The values were acquired from the corresponding differential scanning calorimetry (DSC) curves depicted in **Fig. 3**. In the present study, Table **Table 2** presents the data pertaining to the melting temperature (T_m) and crystalline ratio (X_c) of the PBAT/Zn/PS composites. Meanwhile, **Fig. 4** displays the thermogravimetric analysis (TGA) curves for PBAT/Zn compositions as well as PBAT/Zn/PS blends with 5, 10, and 15 phr of PS. The temperature at 5% weight loss (T_{d5}) and maximum decomposition temperature (T_{max}) were determined by analyzing the corresponding TGA curves and are presented in **Table 3**.

According to a previous study conducted by (Wei et al., 2019), the melting temperature (T_m) of pure PBAT has been determined to be 119.7 °C. Furthermore, based on DSC analysis, with the inclusion of 5% Zein, the melting temperature (T_m) has exhibited an increase to 130.58 °C. The melting temperature (T_m) of PBAT decreases from 130.58 °C to 126.01 °C with the increase in zein content from 5% to 15%. In previous studies conducted by (Botta et al., 2021) and (Chivrac et al., 2006), the melting enthalpy (ΔH^0) of pure PBAT was determined to be 114 J/g. The calculated value of X_c; based on equation 1, in the **Table 2**, which denotes the crystallinity of the PBAT/Zn blends, demonstrated a reduction upon the inclusion of 10% and 15% zein in comparison to the inclusion of 5% zein. The determination of the crystallinity ratio validates the observed trends depicted by the DSC curves. Furthermore, the PBAT/Zn composite curve exhibits a relatively subdued peak, suggesting the presence of an amorphous phase. The absence of a crystallization peak during the heating process indicates that the PBAT/Zn composite can be classified as amorphous. This finding was corroborated by (Corradini et al., 2007), who observed that the plasticized zein with starch displayed no discernible melting characteristics due to its complete amorphous nature.

On top of that, the thermal stability of the PBAT/Zn blend exhibited a marginal improvement compared to the pure PBAT; which depicted 430 °C from (Wei et al., 2019). According to the data presented in **Table 3**, it can be observed that the PBAT/Zn blend exhibited a lower temperature for the 5% weight loss (T_{d5}) point compared to the pure PBAT. The weight loss of 5% associated with 5% zein occurs at a temperature of 368.76 °C (indicated by the red line) which lower than pure PBAT. This weight loss is attributed to the dehydration, depolymerization, and decomposition of thermally unstable proteins and carbohydrate units, as reported by (Thakur et al., 2014; Wei et al., 2019).

Additionally, it is important to mention that the weight of the blend, consisting of 5% zein, undergoes a significant reduction of 60% when the temperature reaches 550 °C. In the current study, it was observed that the degradation process of the sample with a 5% weight loss, containing 10% zein, initiated at a temperature of 358.48 °C. Additionally, it has been observed that the residual weight of the sample amounts to 8% when exposed to a temperature of 550 °C. Nevertheless, it was noted that there was a rise in temperature, as well as an increase in the remaining residue, upon increasing the zein content to 15%. The findings indicate that the incorporation of 10% zein into the blends notably improves the thermal stability of PBAT/Zn blend. As a result, the 90PBAT/10Zn material exhibits a proportional relationship with torque analysis, demonstrating a lower torque value. Therefore, it demonstrates favorable thermal stability properties.

Effect of addition of palm stearin in PBAT/Zein/PS blend

In DSC analysis, the addition of PS to the PBAT/Zn blends resulted in an increase in the melting temperature (T_m) to 133.39 °C. As the concentration of palm stearin (PS) increases from 5 to 15 (phr), the melting temperature (T_m) of the PBAT/Zn blends decreases to 128 °C. In comparison to the other blends, it was observed that the PBAT/Zn/PS15 blend exhibited the highest degree of crystallinity. In previous studies conducted by (Nusantoro, 2009) found that the melting enthalpy of pure PS is 128.4 J/g. The calculated value of X_c; based on equation 1, in the **Table 2**, which denotes the crystallinity of the PBAT/Zn/PS blend, demonstrated a reduction upon the inclusion of PS in comparison to the inclusion of 90PBAT/10Zn. However, it demonstrated an increase with the increasing of PS content. Furthermore, the 90PBAT/10Zn/15PS composite exhibited the highest crystallinity ratio of 17.80%. Higher crystallinity would lead to improved mechanical properties, such as increased stiffness and strength (Beauson et al., 2022). This observation is consistent with the mixing characteristics, which indicate the presence of lower torque values. Consequently, this results in a decrease in viscosity within the blend.

Moreover, the point at which the highest rate of weight loss is observed is referred to as the decomposition temperature, as stated by (Schlemmer et al., 2009). In comparison to the PBAT/Zn blend, it was observed that the thermal degradation (T_{max}) of the PBAT/Zn/PS blends exhibited a slight shift towards lower temperatures. The maximum temperature (T_{max}) of the 90PBAT/10Zn composite was reduced from 440.11 °C to 439.95 °C upon the addition of 5 phr of Palm Stearin (PS). The inclusion of 10 and 15 phr of PS into the PBAT/Zn composite resulted in a decrease in the maximum temperature (T_{max}) to 433.38 °C and 440.05 °C, respectively. The presence of the PS compatibilizer was found to have facilitated the thermal degradation of the polymer composite.

This aligns with the research conducted by (Sudari et al., 2017) on the decomposition of polyethylene (PE) and cellulose. It has been observed that the value of T_{max} decreased from 474.63 °C to approximately 474.17 °C and 467.08 °C upon the addition of hexadecyltrimethylammonium bromide (HTAB) and sodium stearate (SS) to the polymer. However, in contrast, (Eszer & Ishak, 2018) argue that the introduction of Polypropylene-graftedmaleic anhydrides (MAgPP) as a compatibilizer results in a slight increase in the T_{max} of the composite. **Table 3** illustrates that the percentage weight losses have exhibited an increase, thereby indicating a decrease in thermal stability as a consequence of incorporating the PS compatibilizer. The observed phenomenon can be explained by the chemical bond between PBAT and Zein, as well as PS, which potentially compromises the interfacial adhesion. However, it is evident that the 90PBAT/10Zn/10PS blend exhibits superior thermal stability in comparison to other blends, as indicated by its lowest T_{d5} and T_{max} values.

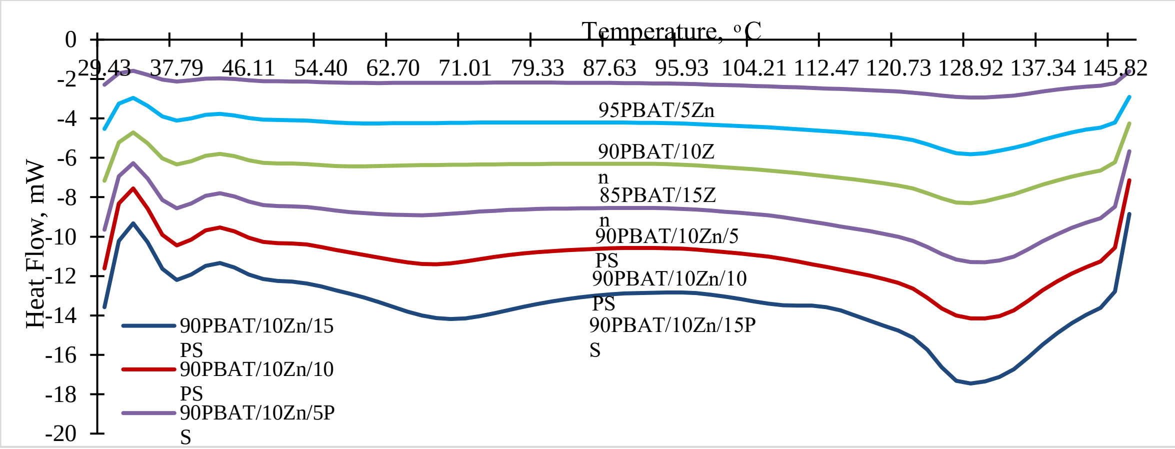

Fig. 3: DSC curves obtained for PBAT/Zn and PBAT/Zn/PS composite blends

Table 2: DSC data of PBAT/Zn and PBAT/Zn/PS blends

PBAT/Zn/PS	PBAT			PS		
	T_m, °C	ΔH_m, (J/g)	X_c, %	T_m, °C	ΔH_m, (J/g)	X_c, %
95PBAT/5Zn	130.58	12.47	218.77	-	-	-
90PBAT/10Zn	128.43	12.99	113.95	-	-	-
85PBAT/15Zn	126.01	4.99	29.18	-	-	-
90PBAT/10Zn/5PS	133.39	15.61	136.93	65.35	5.65	4.63
90PBAT/10Zn/10PS	129.01	11.46	100.53	68.47	11.61	10.05
90PBAT/10Zn/15PS	128	11.23	98.51	71.67	19.43	17.80

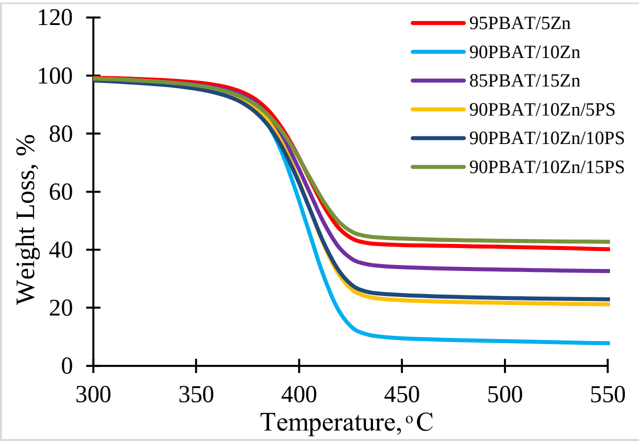

Fig. 4: TGA curves of PBAT/Zn and PBAT/Zn/PS blends

Table 3: TGA data for PBAT/Zn and PBAT/Zn/PS Blends

Compound	T_{d5}, ℃	T_{max}, ℃	Residue (%) (at 550 ℃)
95PBAT/5Zn	368.76	433.5	40
90PBAT/10Zn	358.48	440.11	8
85PBAT/15Zn	365.28	440.07	33
90PBAT/10Zn/5PS	358.23	439.95	21
90PBAT/10Zn/10PS	351.78	433.38	23
90PBAT/10Zn/15PS	363.03	440.05	43

3.3 Effects on the Mechanical Properties

A set of PBAT/zein blends were prepared, with varying zein contents with the presence and absence of PS, by maintaining a consistent blending temperature and molding temperature of 180°C. The tensile strength, elongation at break and modulus of the blend samples, which include varying zein contents with and without PS, are depicted in **Fig. 5** and **Fig. 6**.

Effect of addition of zein in PBAT/Zein blend

With an increase in zein content from 5% to 10%, both the tensile strength and young's modulus demonstrated a significant and rapid increase. However, the addition of 15% zein caused a substantial decline in both the tensile strength (reduced to 2.05 MPa) and the young's modulus (reduced to 87.3 MPa) as depicted in **Fig. 5**. Additionally, the elongation at break also experienced a decrease when 15% zein was incorporated into the blend. Considering the availability of the resulting materials, it was determined that a maximum addition of 10% zein would be suitable for the subsequent experiments. The correlation between the results of the mixing characteristics and thermal analysis indicated that the blend containing 90PBAT/10Zn exhibited the lowest torque value and superior thermal stability compared to the other blends.

Effect of addition of palm stearin in PBAT/Zein/PS blend

Based on the observations, it is evident that a zein content of 10% exhibits the highest performance compared to zein contents of 5% and 15%. However, the addition of PS has adversely affected the overall performance of the blended material. For instance, when 5 phr of PS was added, the tensile strength of the blend decreased from 2.81 MPa (for the blend without PS) to 1.85 MPA. The elongation at break, %, also experienced a decreasing from 3.13 MPa to 1.18 MPa. Conversely, the tensile modulus increased from 105.15 MPa to 132.5 MPa. Additionally, the findings of (Jehsoh et al., 2021) provide support for this claim, as they observed that the NR/HNT composite exhibited optimal mechanical properties with the addition of 0.7 phr of MPS. The observed decrease in mechanical properties following the addition of 0.7 phr of MPS may be attributed to an excessive level of interaction that has occurred. During the mechanical testing, it was observed that the stress concentration point occurred at the interacting point, leading to the development of flaws in the rubber samples. Based on **Fig. 6**, it depicted that the incorporation of PS up to 10 phr has resulted in an enhancement of the tensile stress and young's modulus. However, it should be noted that this particular blend exhibits the lowest elongation at break compared to other blends. The results also demonstrate a correlation with the thermal analysis of TGA, indicating that the blend exhibits superior thermal stability compared to others. This is evident from its lowest T_{d5} and T_{max} values.

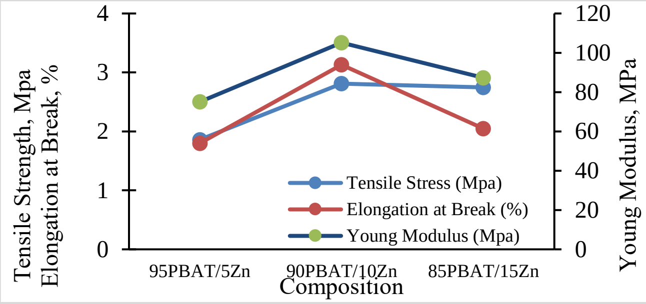

Fig. 5: The tensile strength (MPa), elongation at break (%) and young's modulus (MPa) of PBAT/ZN blend

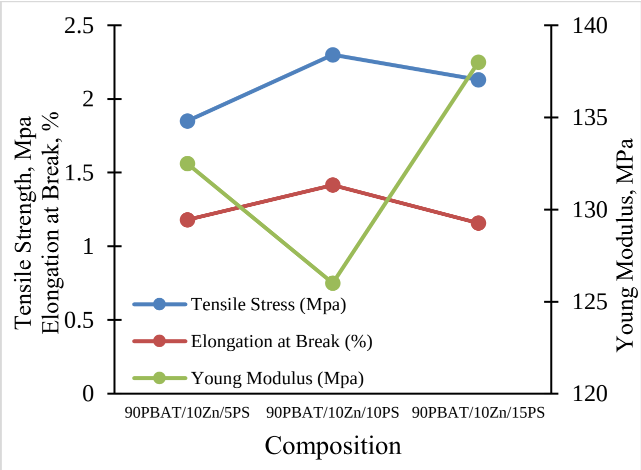

Fig. 6: The tensile strength (MPa), elongation at break (%) and young's modulus (MPa) of PBAT/ZN/PS composites

4.0 Conclusions

In the present study, palm stearin (PS) has been utilized as a compatibilizer for the blend of poly (butylene adipate-co-terephthalate) (PBAT) and zein. The addition of palm stearin (PS) had a minimal effect on the tensile strength, young modulus, and elongation at break of the blends, in comparison to PBAT/zein without PS. After careful analysis, a zein content of 10% (90PBAT/10Zn) has been chosen for the purpose of blending with palm stearin (PS). This decision is based on the superior mechanical and thermal properties exhibited by this blend, in comparison to other blends. The utilization of palm stearin (PS) in the PBAT/Zein/PS blend demonstrated that the 90PBAT/10Zn/10PS composition exhibits superior mechanical and thermal properties when compared to other blends.

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