

Mechanical, Thermal and Chemical Resistance of Epoxidized Natural Rubber Toughened Polylactic Acid Blends

(Rintangan Mekanikal, Termal dan Kimia Adunan Asid Polilaktik Diperkukuhkan dengan Getah Asli Terepoksida)

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ABSTRACT

Polylactic acid (PLA)/Epoxidized natural rubber (ENR-50) blends were prepared by melt extrusion followed by injection molding to fabricate the test samples. The effect of ENR-50 loadings on the morphological, mechanical, chemical resistance and water absorption properties of the blends were studied using standard methods. The toughness of the blend improved with ENR loading up to 20 wt. % but flexural and tensile strength decreased. The balanced mechanical properties were obtained at 20 wt. % ENR-50 loading. SEM showed good distribution and increased ENR particle size as ENR content increased from 10 to 30 wt. %. The differential scanning calorimeter (DSC) showed a steady drop in crystallization temperature (T_c) as ENR content increases while the glass transition temperature (T_g) remained unchanged. Water absorption was observed to increase with ENR loadings. Increase in ENR content was also observed to reduce the chemical resistance of the blends.

Keywords: Blend; epoxidized natural rubber; extrusion; mechanical properties; polylactic acids

ABSTRAK

Adunan asid polilaktik (PLA)/getah asli terepoksida (ENR 50) disediakan dengan menggunakan kaedah penyemperitan leburan dan diikuti dengan kaedah pengacuan suntikan untuk menghasilkan sampel untuk pengujian. Kesan kandungan ENR 50 terhadap ciri morfologi, mekanik, rintangan kimia dan penyerapan air dikaji dengan menggunakan kaedah piawai. Kadar keliatan adunan tersebut meningkat dengan peningkatan kandungan ENR sehingga 20% tetapi kadar kelenturan dan tegangan menurun. Ciri mekanik yang seimbang diperoleh dengan 20% kandungan ENR 50. Imej SEM menunjukkan penyerakan ENR 50 yang baik dan peningkatan saiz partikel selari dengan peningkatan ENR daripada 10% ke 30%. Kalorimeter imbasan pembezaan (DSC) menunjukkan penurunan suhu penghabluran kristal yang stabil dengan peningkatan kandungan ENR 50, manakala suhu peralihan kaca kekal tidak berubah. Kadar penyerapan air didapati meningkat dengan peningkatan kandungan ENR 50. Peningkatan kandungan ENR juga dilihat dapat mengurangkan rintangan kimia adunan tersebut.

Kata kunci: Adunan; asid polilaktik; getah asli terepoksida; penyemperitan; sifat mekanik

INTRODUCTION

At present, most plastics are derived from non-renewable crude oil and natural gas resources. Despite the fact that some plastics can be recycled and reused, majority are disposed in landfills due to end-use contamination. Their disposal raises environmental concerns because of the plastic durability and slow degradation. In some instances, burning the plastics could release toxic fumes. In view of the above the polymer industries face major challenges on how to dispose the plastics after use. To address these problems scientists and researchers have focused their attention on the use of polymeric materials from renewable sources to develop biodegradable materials.

Polylactic acid (PLA), a biodegradable polymer with high biocompatibility and good mechanical properties is one of the promising bio-based materials currently available in the market (Ishida et al. 2009). Its biodegradability is

an asset in application areas that recycling is difficult. Therefore, the production of PLA from mainly starch and sugar has gained attention as an alternative to conventional synthetic polymers.

Studies have shown that PLA is similar to poly (ethylene terephthalate) (PET) in many ways. PLA is a rigid and transparent material with low crystallization rate. This makes it a good material for the fabrication of stretch-blown bottles, biaxially oriented films and thermoformed containers (Huneault & Hongbo 2007). PLA has become a replacement for PET and PS in many traditional application areas such as foamed food trays, fibers, thermoformed packaging and card stock. It is also used in blends with thermoplastics and other less conventional applications such as housing for computers and electronics.

The major drawbacks which reduce the use of PLA as a plastic material in many applications are its stiff and brittle

nature. These properties results in the low impact strength and deformation at break exhibited by PLA (Jacobsen et al. 1999). In view of the above, PLA is blended with other polymers to impart flexibility and toughness and also to reduce its cost as it is expensive as compared to other conventional polymers.

Ho et al. (2008) reported that the toughening of PLA can be achieved when it is grafted to maleic anhydride-functionalized thermoplastic polyolefin (TPO-MAH) in the presence of 4-dimethylaminopyridine. The elongation at break and tensile toughness were enhanced due to improved interfacial interaction induced by the TPO-PLA copolymer compatibilizer. However, the tensile strength and tensile modulus dropped due to the elastic behaviour, low strength and modulus of the system.

The effect of dicumyl peroxide (DCP) as a cross linking agent in blends of poly(lactic acid)/poly(ϵ -caprolactone), (PLA/PCL) has been investigated. The researchers reported improvement in the mechanical properties after the incorporation of the DCP. Based on their DMA, melting interfacial tension and tensile test results, they submitted that the blends were compatible when small quantity of DCP is added. They concluded that DCP is a good compatibilizer for the PLA/PCL blend (Takeshi et al. 2006).

PLA has also been blended with other rubber materials to enhance its properties. The researchers reported yields of spherical rubber particles which act as stress concentrators in the blend thus, initiating and terminating crazes in the brittle polymer matrix (Ishida et al. 2009). The initiation and termination of the crazes results in increase in the fracture energy absorption of the system thereby improving its toughness.

In this research, PLA is blended with epoxidized natural rubber (ENR-50) as an impact modifier to improve its toughness. The effects of the ENR-50 contents on the morphological, mechanical, thermal, water absorption and chemical resistance were successfully studied using the various techniques.

EXPERIMENTAL DETAILS

MATERIALS

PLA is a commercial product from Biomer, Kraling, Germany. It has average molecular weight, $M_{wt}=220$ kDa; $M_n=101$ kDa with density, 1.25 g/cm^3 . The ENR-50, 'Epokrena 50' was supplied by the Malaysian Rubber Board. It has 50% epoxidization level.

PREPARATION OF BLENDS AND TEST SAMPLES

The ENR-50 was masticated using two roll mills after which it was cut into small pieces. The pieces were continuously added to the PLA during the extrusion process. The PLA pellets were dried in a hopper drier at 40°C for 24 h prior to compounding to remove the moisture. The Brabender Plasticoder PL 2000 counter-rotating twin-screw extruder was used to compound the

blends at temperatures between 190°C feed zone and 175°C at die head and 40 rpm screw speed to achieve enough shear mixing. The extruded blends were air-dried, pelletized and stored for injection moulding. Prior to injection moulding, the pellets were dried at 40°C for 24 h in an oven. The injection moulding was carried out using JSW (Muroran, Japan) Model NIOOB II injection moulding machine. The barrel temperature ranged from $165\text{-}190^\circ\text{C}$. The fabricated specimens were stored in desiccators for 48 h prior to testing. Table 1 shows the blend compositions and designation of materials.

TABLE 1. Blends composition of PLA/ENR-50

Composition	Designation	Mixing ratio (wt.%)	
		PLA	ENR-50
PLA	PLA	100	0
PLA/ENR-50	P90/E10	90	10
PLA/ENR-50	P80/E20	80	20
PLA/ENR-50	P70/E30	70	30

TESTING METHODS

TENSILE TEST

The standard specimens were tested using the Instron (Bucks, UK) 5567 tensile testing machine according to ASTM D638 at a crosshead speed of 5 mm min^{-1} at ambient temperature. Five samples were tested and the average value was reported.

FLEXURAL TEST

This test was carried out using the AG-5kNE Shimadzu universal testing machine according to ASTM D790. The test was done under ambient conditions at a crosshead speed of 3 mm min^{-1} . Five standard specimens were tested and the average value was reported.

NOTCHED IZOD IMPACT TEST

In order to determine the toughness of the blends, the samples were notched and tested using the Toyoseiki (Tokyo, Japan) impact testing machine according to ASTM 256. The samples were notched using an automatic notching machine prior to testing. Five specimens of each formulation were tested and the average value was reported.

MORPHOLOGICAL STUDIES

SCANNING ELECTRON MICROSCOPY (SEM)

The morphology of PLA and PLA/ENR blend was studied using the scanning electron microscope (JEOL JSM-6390 LV Model). The specimens were etched with xylene for 3 h after which they were fractured. The samples were

obtained from the fractured surfaces and dried in a vacuum oven at 60°C for 4 h before coating with gold. The coated specimens were mounted on sample stubs using double sided adhesive tapes and then evaluated using the SEM.

THERMAL ANALYSIS

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

The melting and crystallization behaviour of the blends were studied using Perkin-Elmer differential scanning calorimetry after calibrating the temperature with Indium. 5-8 mg of each sample was sealed in aluminium pan and used for the DSC measurement. Temperature was raised from 25 to 200°C at a heating rate of 10°C/min. The percentage crystallinity, X_c was calculated using (1):

$$\% \text{ Crystallinity, } X_c = \frac{\Delta H_m}{\Delta H_m^0} \times \frac{100}{w}, \quad (1)$$

where ΔH_m is the melting enthalpy; ΔH_m^0 is the melting enthalpy for 100% crystalline PLA sample taken as 93.1 J/g (Abdelwahab 2012); and w is the weight fraction of PLA in the blend samples.

THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermogravimetric analysis was performed using the Mettler Toledo model TGA/SDTA851 thermal analyzer. Determinations were carried out at temperatures ranging from 30 to 900°C at heating rates of 10°C/min under nitrogen atmosphere.

CHEMICAL RESISTANCE

The chemical resistance test was carried out according to ASTM D543-06. The changes in mass and appearance of PLA/ENR-50 blends were investigated after immersing the specimens in engine oil (4t Honda motor oil, SJ 10W3) and palm olein cooking oil (Vesawit). The samples were suspended in appropriate containers to avoid contact with the walls or bottom of the container and then filled with oils until the samples were totally immersed. After 24 h of immersion in the oils, the samples were withdrawn and rinsed with water. They were then wiped with tissue paper to remove the leftover oils. The samples were then weighed and their weight reported to the nearest 0.001 g. The procedure was repeated for 96 and 168 h. The tests were carried out in triplicates for each blend formulation and the average value was reported. The weight difference was calculated using (2).

$$\text{Percentage weight difference (\%)} = \frac{(m_2 - m_1)}{m_1} \times 100\%, \quad (2)$$

where m_1 is the weight before immersion; and m_2 is the weight after immersion.

WATER ABSORPTION

The water absorption test was conducted to determine the water resistance of PLA and PLA/ENR-50 blends. Test specimens of dimensions 30 × 5 × 3 mm were dried in a vacuum oven at 40°C until they attain a constant dry weight. The specimens were then immersed in distilled water at room temperature for 24 h. After 24 h, the samples were withdrawn from the water, wiped with tissue paper and then weighed. The weight gain after 24 h was recorded. The procedure was repeated with samples immersed for 96 and 168 h. The test was carried out in triplicates for each formulation and the average value was reported. The percentage weight gain, M_t after time, t of moisture absorption was determined using (3).

$$M_t (\%) = \frac{(W_w - W_d)}{W_d} \times 100\%, \quad (3)$$

where W_d is the initial weight of samples before immersed in water; and W_w is the weight of samples after water immersion.

RESULTS AND DISCUSSION

MECHANICAL PROPERTIES

Figure 1 illustrates the tensile strength and Young's modulus of PLA/ENR-50 blends. Tensile strength decreased from 12.05 to 5.91 MPa as ENR-50 content increased from 10 to 30 wt. %. This is 67% drop in tensile strength as compared to the virgin PLA. The decrease in tensile strength was attributed to the toughening effect of ENR-50 as earlier reported by Akbari et al. (2013) and Mohanty et al. (1996). The Young's modulus was also observed to decrease from 1.38 to 1.04 GPa at 10 to 30 wt. % ENR-50 content. This is 58% reduction in Young's modulus as ENR-50 was incorporated into PLA. The drop in Young's modulus was attributed to the low modulus of the ENR-50. Similar observations have been reported by Chuayjuljit et al. (2006), Ishida et al. (2009) and Tanrattanakul et al. (2008).

The effect of ENR-50 content on the flexural properties of PLA/ENR-50 blends is shown in Figure 2. The flexural strength was observed to decrease sharply from 90 to 7 MPa as ENR-50 content increased from 10 to 30 wt. %. This was attributed to the drop in stiffness of the blend as earlier observed. Also, at high ENR-50 content large ENR particle sizes are formed thus contributing in lowering the flexural properties of the blend (Figure 6(d)). Similar observations have been reported by Ruamcharoen et al. (2011). The flexural modulus decreased steadily from 3.5 to 1.7 GPa with increasing ENR-50 content. At 30 wt. % ENR-50 loading, the flexural modulus of the blend decreased by 106%. The sharp decrease in the flexural modulus was also attributed to the low modulus of the ENR-50.

Figure 3 is an illustration of the effect of ENR-50 loading on the impact strength of PLA/ENR-50 blends.

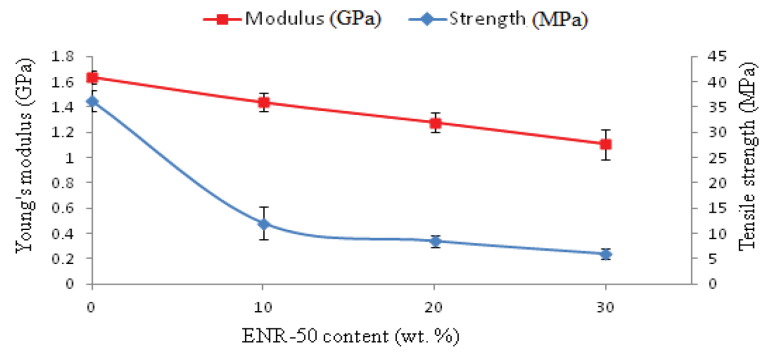


FIGURE 1. Effect of ENR-50 content on tensile strength and Young's modulus of PLA/ENR blends

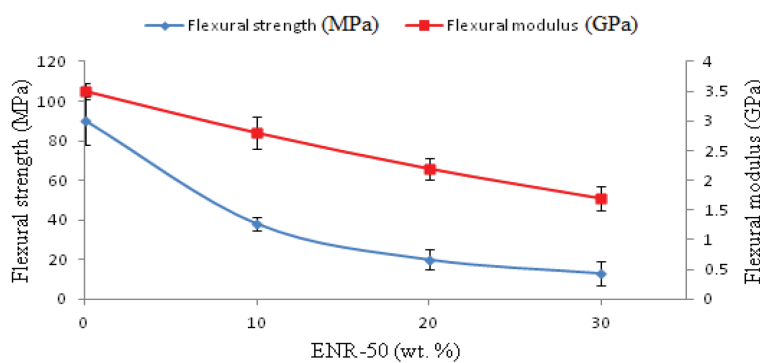


FIGURE 2. Effect of ENR-50 content on flexural strength and modulus of PLA/ENR-50 blends

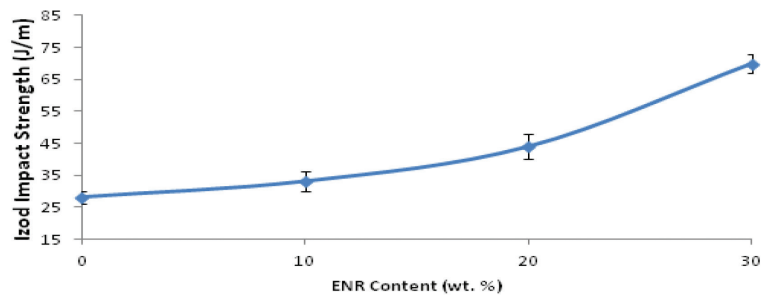


FIGURE 3. Effect of ENR content on the Izod impact strength of PLA/ENR blends

Impact strength increased from 33.15 to 69.82 J/m as ENR content increased from 10 to 30 wt. %. The increase is attributed to the ENR addition. The ENR-50 which acts as an impact modifier improves the toughening and ductility of the PLA thus increasing the energy absorption capacity of the blends. ENR-50 when well dispersed in a blend acts as an effective stress concentrator thereby enhancing crazing and shear yield in the blend. Thus the 30 wt. % ENR blend exhibited the highest impact strength among all the formulations. Similar observations have been reported by Borggreve et al. (1987). Low density cross links which have been proposed to exist (Yew et al. 2005) in PLA/ENR-50 blends also contribute in the enhancement of stress

transfer between the brittle PLA phase and the tough ENR-50 matrix thus increasing the impact strength (Ahmad Sharif et al. 2011). The dissipation of impact energy through multiple crazing by the ENR-50 is another reason given for the increase in impact strength of the blends as ENR-50 content increases (Bucknall 1977). This is supported by the SEM micrographs shown in Figure 6.

Figure 4 is an illustration of the elongation at break for the PLA/ENR-50 blends. The elongation at break dropped as ENR-50 content increased in the blend despite the fact that ENR-50 has greater elongation than PLA. This was not expected as increase in ENR-50 content resulted in impact strength to increase. The reduction in elongation at break

was attributed to interactions between the PLA and the ENR-50 chains as earlier proved by Akbari et al. (2013) and Mishra et al. (2007).

BALANCED MECHANICAL PROPERTIES

The attainment of balanced mechanical properties in materials developed and processed is important in the field of engineering. In view of the above, obtaining balanced stiffness and toughness is a cardinal objective in the mechanical property performance of a material. Figure 5 shows the effect of ENR-50 content on the stiffness and toughness of PLA/ENR-50 blends. Previous discussions have shown that the incorporation of ENR-50 improved the toughness of PLA/ENR blends at the expense of stiffness and strength. Figure 5 shows that the formulation with the balanced mechanical properties is PLA80/ENR20 which has a flexural modulus of 2.8 GPa and impact strength of 47 J/m.

MORPHOLOGICAL STUDIES

Figure 6 shows the SEM micrographs of the impact fractured surfaces of neat PLA and blends of PLA/ENR-50. The smooth brittle fractured surface of neat PLA is shown in Figure 6(a). The good dispersion of ENR-50 in the 10 wt. % PLA/ENR-50 loaded blend is shown in Figure 6(b). The black holes represent the ENR-50 particles etched by xylene.

The micrograph also shows homogenous and uniformly dispersed ENR-50 particles. Figure 6(c) is the micrograph of the fractured surface of the blend at 20 wt. % ENR-50 content. The micrograph showed a coalesce morphology of larger particle size as compared to the 10 wt. % ENR-50. The large particle sizes as compared to the 10 wt. % ENR-50 resulted from the breakup and coalescence of the ENR-50 during blending and also the increase in concentration of the ENR-50. Similar findings have been reported by Ishida et al. (2009). Figure 6(d) shows larger particles of ENR-50 in the blend at 30 wt. % loading thus confirming the increase in particle size as ENR content increases in the blend.

THERMAL ANALYSIS

Table 2 shows the glass transition temperature (T_g), crystallization temperature (T_c) and melting temperature (T_m) obtained from the differential scanning calorimetry (DSC) analysis for PLA and PLA/ENR blends. The repeated heating and cooling process effectively removed the thermal history of the system. This is confirmed by the absence of crystallization peaks during cooling and also the fast quenching of samples which prevented crystallization. Similar findings have been reported by Calvão et al. (2010).

The PLA had melting temperatures between 164 and 167.5°C and a crystallization exotherm which start at about 93.7°C and ends at 100.8°C. The T_g for PLA in

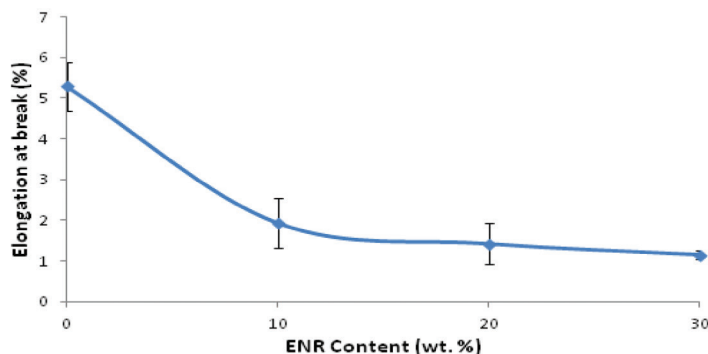


FIGURE 4. Effect of ENR content on elongation at break of PLA/ENR blends

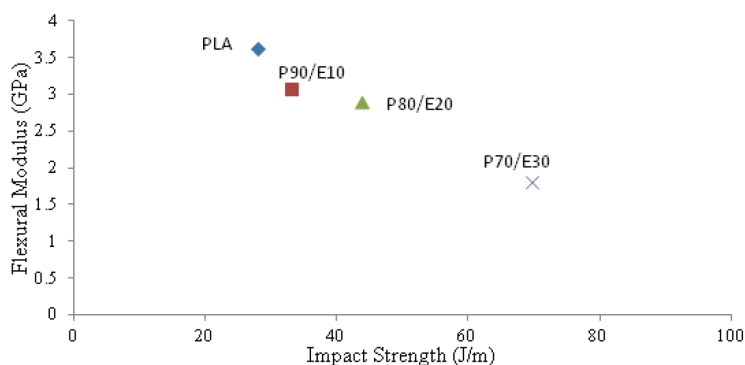


FIGURE 5. Balanced mechanical properties based on flexural modulus and impact strength of PLA/ENR blends

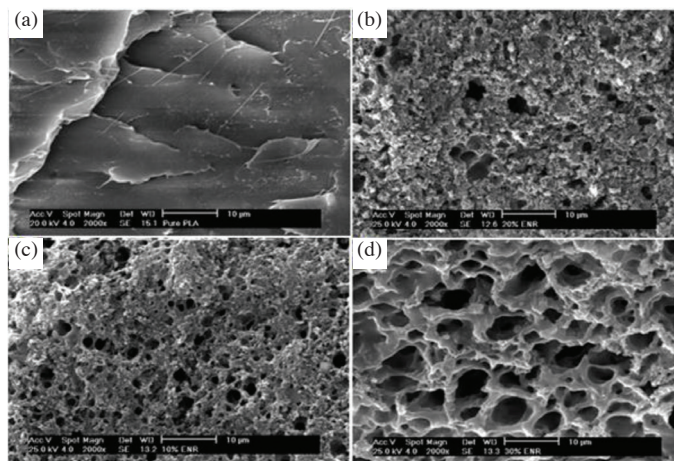


FIGURE 6. SEM micrographs of PLA/ENR blends (a) pure PLA, (b) PLA90/ENR10, (c) PLA80/ENR20 and (d) PLA70/ENR30

TABLE 2. Thermal characteristics of PLA and PLA/ENR blends

Composition	T_g (°C)	T_c (°C)		T_m (°C)	X_c (%) PLA
		Heating	Cooling		
PLA	65.0	100.8	Not observed	167.5	41.6
P90/E10	65.4	95.4	Not observed	166.7	47.1
P80/E20	64.9	94.3	Not observed	164.5	48.0
P70/E30	65.2	93.7	Not observed	164.0	51.6

the blends is approximately the same as that of the neat PLA (65°C). This suggests that the PLA/ENR blends were immiscible. This agrees with previous findings reported by other researchers (Ishida et al. 2009). The T_c of PLA was observed to decrease as ENR content increases in the blend. This was attributed to the possibility of the ENR acting as nucleating sites for the PLA especially when the ENR is well dispersed in the PLA. Nucleating effect leads to decrease in T_c and increase in the crystallinity of the PLA/ENR-50 blends. Similar findings have been reported by Calvão et al. (2010).

Figure 7 shows the TGA curves for the thermal degradation of PLA/ENR blends loaded with 10 to 30 wt. % ENR-50. The thermal stability of the neat PLA and PLA/ENR-50 blends were characterized based on the temperature at which 10% (designated as T_{10}) weight losses were recorded and presented in Table 3. Two step degradation processes were observed for all the PLA/ENR-50 blends. The variations of T_{10} for all the blends are summarized in Table 3. The thermal stability of the blends reduced as ENR-50 content increased from 10 to 30 wt. %. This was attributed to the presence of the oxirane

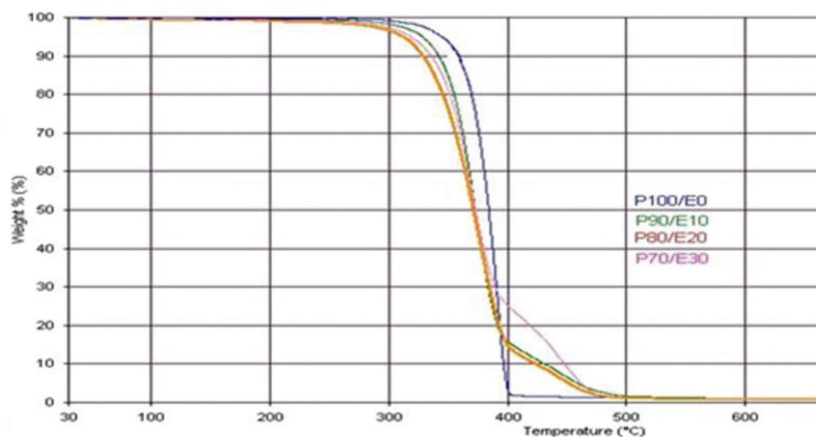


FIGURE 7. Thermogravimetric analyses of PLA and PLA/ENR with different ENR contents

TABLE 3. TGA results of PLA and PLA/ENR blends

Composition	ENR content (wt%)	T10%(°C)	ENR residue at 400°C
PLA	0	357.4	3.4
P90/E10	10	341.8	15.9
P80/E20	20	328.7	15.0
P70/E30	30	335.6	25.2

groups in the ENR-50. At high temperatures, oxidation of the epoxide group in PLA/ENR-50 blend takes place. Also, ENR on its own can act as an auto oxidant by initiating free radical reaction. The free radicals generated subsequently initiate the oxidation of the PLA in the blend.

CHEMICAL RESISTANCE

The chemical resistance of PLA/ENR blends to engine oil and cooking oil (palm olein) are shown in Figures 8 and 9. The swelling effect after 24, 96 and 168 h of immersion in engine oil is shown in Figure 8. A steady increase in weight was observed for all the blends after immersion for 24 h. The PLA/ENR-50 blend with 30 wt. % ENR content exhibited the highest swelling. This signifies that ENR-50 reduced the blends capacity to resist the absorption of engine oil. The high engine oil absorption was attributed to its high polarity. Also, the degree of epoxidation of ENR is believed to determine the polarity of ENR. As epoxidation increases,

the ENR viscosity increases thereby increasing the polarity of the ENR. Similar findings have been reported by Baker and Gelling (1987). This implies that as ENR-50 is polar, it will enhance the absorption of the polar engine oil as its quantity increased from 10 to 30 wt. % in the blend.

Figure 9 is the swelling effect of the blends after immersion in palm olein cooking oil for 24 to 168 h. A steady increase in weight was also observed for the blends after immersion for 24 h. The PLA/ENR-50 blend with 30 wt. % ENR content had the highest swelling. This signifies that ENR-50 reduced the blends capacity to resist absorption of the engine oil. It is worth to take note that the absorption of palm olein cooking oil by the blend was less as compared to the engine oil. Comparing the 30 wt. % PLA/ENR-50 blend, 18% increase in weight was recorded after 168 h of the blend immersion in engine oil as against 6% weight gain for those immersed in palm olein oil. This indicates that engine oil has higher polarity as compared to palm olein cooking oil (Kiattanavith 1986).

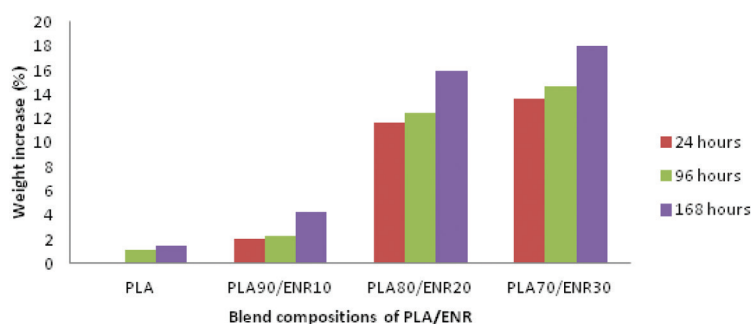


FIGURE 8. The swelling effect of PLA/ENR blends after immersion in engine oil

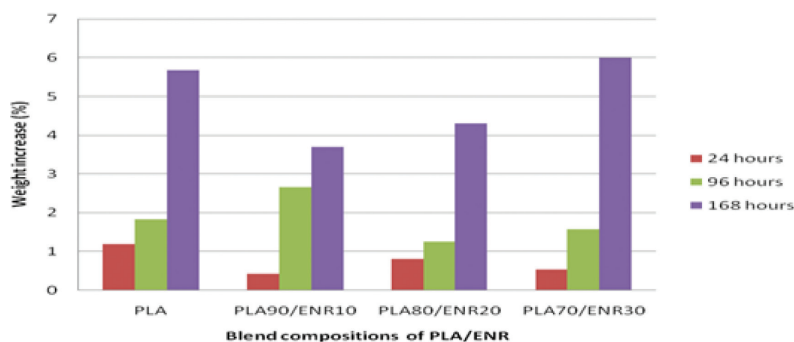


FIGURE 9. The swelling effect of PLA/ENR blends after immersion in palm olein cooking oil

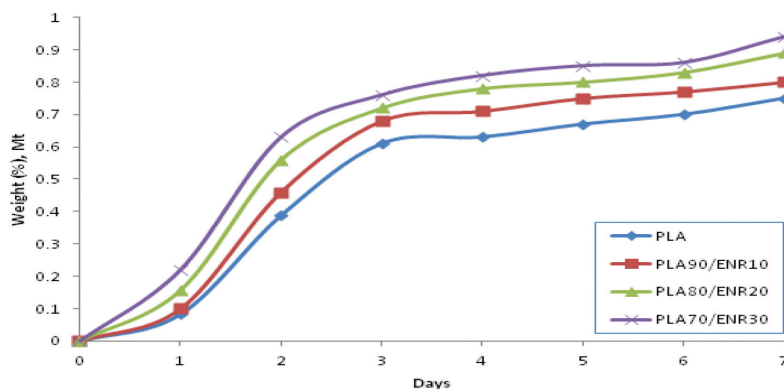


FIGURE 10. Water absorption curves of pure PLA and PLA/ENR blends

WATER ABSORPTION

Figure 10 shows the water absorption curves for pure PLA and the PLA/ENR blends. Incorporating ENR-50 increased the water uptake of the blends. The increase in water absorption increased as ENR-50 content increased from 10 to 30 wt. %. It was also observed that all formulations of the blend gave higher water absorption values than that of pure PLA. The increase in water absorption was attributed to the hydrophilic nature of the ester group of PLA and the oxirane group present in ENR-50. The ENR-50 being polar materials enhances hydrogen bond formation with water thus increasing water absorption (Yew et al. 2005). The absorption of water molecules within the short period of immersion was attributed to the exposed ENR-50 molecules on the surface of the blends. The increase in ENR-50 in the blend implies a favourable condition for more water molecules to be absorbed into the system. As the surface particles absorb more water molecules, they become saturated thus encouraging further penetration of the water molecules into the blends. Garlotta et al. (2003) have reported similar findings.

CONCLUSION

PLA/ENR-50 was successfully blended to improve the toughness of the PLA. The impact strengths of the blend increased with increasing ENR-50 content. The PLA/ENR blends containing 20 wt. % of ENR-50 content had the best balanced mechanical properties.

The DSC results showed increase in crystallinity as ENR-50 content increases while the crystallization temperature dropped. The thermal stability of the blends dropped with the increase in ENR-50 loading. The PLA/ENR-50 blends were observed to generally exhibit poor chemical and water resistance as ENR-50 was incorporated into the system.

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Received: 28 March 2015

Accepted: 12 June 2015