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Original Research Article

Effect of Maleated Polyethylene Wax on Mechanical and Rheological Properties of LDPE/Starch Blends

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ABSTRACT

The non-biodegradable behaviour of polyethylene causes a serious environmental hazard related to the increasing volume of plastic waste. Therefore, the blending of polyethylene with biodegradable polymers like starch enhances the polyethylene film biodegradation rate. This study was aimed at investigating the compatibility of low-density polyethylene (LDPE)/plasticized starch (PLST) blends using maleated polyethylene wax (m-PEwax) as compatibilizer. M-PEwax was prepared, and different amounts of the compatibilizer were added to the LDPE/PLST blend. FTIR spectroscopy was used to analyze the blends. Mechanical and rheological properties of blends were determined. The FTIR results indicated that maleic anhydride was successfully grafted onto polyethylene wax (PEwax), and also clearly showed the interface in the LDPE/PLST blends that contain m-PEwax. The mechanical properties revealed a reduction in the tensile strength, break energy, and slightly increased the elongation at break. However, increasing the ratio of m-PEwax >8 wt% caused phase separations. Both the shear stress and rate increased. Meanwhile, at a given shear rate, the shear stress was reduced slightly along with the ratio of m-PEwax. The rheological measurements also proved that m-PEwax decreased the viscosity and enhanced the fluidity of LDPE/PLST blends. The flow index (*n*) of the blends increased as the m-PEwax content increased. Moreover, a reduction in the activation energy led to an increase in the m-PEwax content. Our findings revealed the presence of m-PEwax in the LDPE/PLST blend, making it a potential candidate for partial manufacture of biodegradable film which might be used as agricultural mulch or for a variety of packaging applications.

Keywords: Compatibilizer, LDPE/PLST blend, Maleated polyethylene wax, Mechanical properties, Rheological properties.

Introduction

Polyethylene is a non-biodegradable polymer. It is widely used as a commodity thermoplastic material in disposable packaging and many other industrial applications. Therefore, there is a growing interest in ensuring that the products are biodegradable from the environment.[1,2] A mixture of biodegradable and non-biodegradable polymers is more benefical than manufacturing entirely biodegradable polymers as the latter has economic advantages and superior properties.[3,4] Starch and cellulose derivatives are preferable because they biodegrade easily in a soil environment. Accelerated biodegradation in the environment of polyolefinic wrapping materials has been achieved through the use of widely available polyethylene blends with natural products, particularly starch.[5-7] Chemically, starch is an hydroglucose polymer ($C_6H_{10}O_5$). It consists of 17-27 wt. % amylase with α-1,4-links between the units and α-1,6-linked amylopectin (amylose is a linear polymer).[8] Starch is mechanically brittle and difficult to thermally process with other polymers due to the strong inter-and intra-molecular hydrogen bonds among the glucose macromolecules in starch. Plasticizers, such as glycerol, can

be used to modify starch at high temperatures, reducing connections between molecule chains and breaking the starch structure.[9]

In polyethylene and starch blends, microbes initially invade the starch resulting in a more porous structure that lowers the surface-to-volume ratio which improves its biodegradability.[10] Low-density polyethylene (LDPE) and starch are immiscible because LDPE is hydrophobic and starch is hydrophilic. There are many ways to enhance the miscibility between these two polymers. The miscibility can be improved by enhancing the starch hydrophobicity,[11] or enhancing the LDPE hydrophilicity.[12] Another method to make suitable polymer blends is to mix reactively *in situ* or to use a compatibilizer that contains maleic anhydride (MAH).[13,14] The anhydride group can graft other polymers at the melt temperature due to its high reactivity in subsequent processes. Bikiaris et al., found in their study conducted in 1998 that polyethylene-g-maleic anhydride co-polymers (PE-g-MAH) was the most effective compatibilizers in LDPE/ plasticized starch (PLST) blends.[15,16] Nevertheless, these blends are expensive and hard to make, and their rate of biodegradation is about the same as that of incompatible blends.

Polyethylene wax (PEwax) is a by-product of the LDPE plant of the petrochemical company in Basrah, Iraq. It has a chemical structure that is similar to LDPE. As a result, PEwax miscibility within the LDPE matrix is excellent.[17] Also, it is used for soil microorganisms as the sole source of carbon and energy and can be degraded by bacteria and fungi residing in the soil.[18-20] Therefore, using PEwax grafted with MAH would increase the level of biodegradation and lower the cost of the LDPE/starch blends. PEwax is a relatively new blend of biodegradable blends and has not yet been widely documented. Rosa *et al*.,[21] have shown in their 2007 study that the effect of oxidized PEwax on the biodegradation properties of polyβ-

hydroxybutyrate/LDPE blends could minimize phase separation in the blend between the two polymers and slightly increase the rate of biodegradation in simulated soil. In 2009, Chuayjuljit et al. [22] used PEwax as a processing aid in LDPE/Starch/modified montmorillonite nanocomposites. Maleated PEwax (m-PEwax) was prepared in this study and used as a compatibilizer of LDPE/PLST blends. The aim of the study was to investigate the effect of maleated polyethylene wax on the mechanical and rheological properties of LDPE/starch blends.

Materials and Methods

Sources of chemical materials
Low-density polyethylene (LDPE; density: 0.921–0.924, melting index: 0.28–0.38) and polyethylene wax (PEwax; an average molecular weight: ~2560±20) were obtained from Petrochemical Industry Company, Basrah, Iraq. Starch was supplied by Merck Company. Chemicals Fertilizer LTD, India, provided MAH, while Fluka supplied glycerol and azobisisobutyronitrile (AIBN). The maleated polyethylene wax (m-PEwax) was made with a grafting percentage of about 0.4 % in the Chemistry Department laboratory, University of Basrah, Basrah, Iraq. The starch was plasticized with 10 % glycerol and is referred to hereafter as PLST.

Preparation of maleated polyethylene wax (m-PEwax)
Maleated polyethylene wax (m-PEwax) was prepared by heating and melting 34 g of PEwax in the coin container. Afterward, 4 g of MAH was added dropwise to 2 g of molten PEwax. AIBN was added at 2 min in four portions. The mixing was continued for another 2 minutes after the last addition. After the dropwise addition was completed, the stirring was continued. The reaction was stopped after the desired time was reached, and the product was washed with acetone several times to remove the unreacted MAH.[23] To determinate the grafted MAH in m-PEwax, 1 g of PEwax was heated for hydrolysis of anhydride groups in 150 ml refluxing water-saturated xylene. Using 3-4 drops of 1 % thymol blue in DMF as an indicator, the hot solution was immediately titrated with 0.05 N ethanolic KOH. An aliquot of 0.5–1.0 ml KOH solution excess was applied, and by adding 0.05 N isopropanol HCl to the hot solution, the deep blue color was back-titrated to a yellow endpoint. The number of acids and the grafted MAH content was calculated,[23] as follows:

$$Acid\ number \left(\frac{mg\ KOH}{g\ Polymer}\right) = \frac{mL\ KOH \times N\ KOH \times 56.1}{polymer\ (g)}$$

Therefore,

$$MAH\ \% = \frac{Acid\ number\ \times 98}{2\ \times 56.1}$$

Preparation of blends
In a Haake Rheomix (Model 600) with rollerblades and a 60 cm^3 volumetric mixing handle, LDPE was dried in an oven for 2 hours at 80 °C and melted with the PLST. The materials were then carefully premixed in correct amounts. Mixing was carried out for 10 min at 150 °C, and 50 rpm. Five different weights of m-PE wax namely 2, 4, 6, 8, and 10 wt % with 20 wt % of PLST of the total weight of these blends, were used for each combination of LDPE/PLST blends.[15,16] The blends were milled and put in the mold after preparation using a hydraulic press and stored for 10 min at 20 °C. After that, the mold was cooled in water until it reached room temperature. The mold was then removed, and the material was slapped and cut into a dumbbell shape for mechanical property testing. LDPE/PLST blends without the compatibilizer were also prepared for use as reference material.[17]

FTIR spectroscopic analysis of blends
Using a Fourier Transform Infrared (FTIR) 8400S spectrometer (Shimadzu, Japan), FTIR spectra were obtained. The samples were determined for each spectrum in the form of thin films prepared by hot-press molding.

Measurement of mechanical properties of blends
The mechanical properties, such as tensile strength and elongation at break, were measured using a tensile instrument (Elma, Tehran, Iran) according to the ASTM D882 method. The measurements were made using a crosshead speed of 20 mm/min. From each sample, five dumbbell shapes were cut and the measurements were summed to get a mean value.[7,9]

Determination of rheological properties of blends
To determine the rheological properties of all blends, a capillary rheometer (Instron model 3211) was used. The capillary die measurements were 1.257 mm, the ratio of length to diameter (L/D) was 80/9, and the angle of entry was 90°. The rheological data were collected at various temperatures (150, 160, 170, and 180 °C) for all blends over shear levels of 5.43, 18.1, 54.3, 181, 543, and 1810 s^{-1}. The samples were stored in the rheometer's temperature-controlled barrel and pushed through a properly dimensioned die by a piston. The shear rate delivered to the material was determined by the die geometry and piston velocity. To measure the shear stress and hence the viscosity, the pressure at the entry to the die was used. Below is a brief review of the equations used in a capillary rheometer to measure shear stress, shear rate, and viscosity. Equation 1 was used to measure the shear stress on the wall and Equation 2 was used to calculate the shear pressure on the wall.[7]

$$Wall\ shear\ stress\ (\tau_w) = 3.44 \times 10^5 \times F \times \frac{dc}{L}\ldots\ldots\ldots (1)$$

Where F is the piston force in Kg, L is the capillary tube length, and d_c is the cylinder radius.

$$Wall\ shear\ rate\ (\gamma_w) = 0.133 \left[\frac{3\eta+1}{4\eta}\right] V_{XH}\left(\frac{d_b^2}{d_c^3}\right)\ldots\ldots\ldots (2)$$

Where V is the velocity of the fluid flowing through the capillary tube and d_b is the cylinder diameter. Equation 3 was used to calculate the apparent viscosity:

$$\eta_a = \frac{\tau_w}{\gamma_w}\ldots\ldots\ldots (3)$$

Statistical analysis
Data obtained from the study were expressed as mean ± standard error of mean (SEM). The statistical analysis was performed using the SPSS software (version 20). One-way ANOVA was used for comparing means and a p value of <0.05 was considered significant.

Results and Discussion

FTIR spectra of polyethylene and maleated polyethylene waxes
The FTIR spectra of polyethylene wax (PEwax) and maleated polyethylene wax (m-PEwax) are shown in Figure 1. When comparing the m-PEwax spectra to that of pure PEwax, stretching vibration bonds (symmetrical and asymmetrical) of maleic anhydride groups were observed at 1853 and 1778 cm^{-1}, respectively.[23] Besides the characteristic bands of maleic anhydride groups, the absorption occurred around 1711 cm^{-1}, which is consistent with the appearance of C=O bonds from the hydrolysis reaction of the anhydride group.[13] The FTIR spectrum of pure LDPE (Figure 2) revealed strong intensities for methylene vibration at rang 2851–2953 and 724 cm^{-1} as well as a peak at 1463 cm^{-1} which was attributed to bond vibration of –CH$_2$– and methyl branching at 1366 and 1302 cm^{-1}. These findings show the presence of minor branching of the chain in LDPE.[24] In the spectrum of PLST in Figure 2, the broad peak in the region 3075-3600 cm^{-1} was due to the hydroxyl groups of the glycerol present in the PLST and the hydroxyl groups of the glucose units in the starch. Three peaks at 928, 994, and 1249 cm^{-1} appeared in the spectrum due to C-O-H vibration bond stretching.[25]

In the FTIR spectrum of LDPE/m-PEwax/PLST blend as presented in Figure 1, the bold line shows the disappearance of maleic anhydride peaks at 1853, 1778, and 1711 cm^{-1} and formed a sharp peak at 1729 cm^{-1}; this peak is reverted to ester groups which resulted from the reaction of PLST hydroxyl groups with the maleic anhydride groups. Thus, the blends' FTIR measurements showed that the maleic anhydride groups of the m-PEwax reacted with the hydroxyl groups of PLST. This result implies an enhancement in the compatibility of blends in the presence of m-PEwax and agrees with the findings of other studies when maleated polyethylene was used as compatibilizer.[15,16]

Mechanical properties of blends

Table 1 shows the results of the stress-strain curve plots of LDPE/PLST (100/20 wt %) blends and the corresponding data for tensile stress at both yield and break, elongation and break energy. The LDPE/PLST blends without compatibilizer have acceptable tensile strength (about 8.34 MPa), which was reduced to 16.53 % compared to that of pure LDPE (10.1 MPa), due to good homogeneous and fine dispersion of PLST in the LDPE matrix. [26] For the LDPE/m-PEwax/PLST blends, the tensile strength typically decreased with increasing m-PEwax content of the blend until the ratio reached 8 wt %. However, when the content of m-PEwax was 10 wt%, the mechanical properties decreased significantly because the amorphous areas in the LDPE were saturated and inadequate for m-PEwax accommodation. [17] Generally, m-PEwax had short-side hydrocarbon and maleic anhydride homopolymer chains which led to a loss of regulation in the polymer blend chains and reduced crystallinity. As a result, these branch chains had different modes and were irregular, which could have reduced the tensions between the molecular chains, resulting in a gradual loss in tensile strength at yield and break, as well as break energy. [27,28]

The elongation at break can also be calculated based on the stress-strain curves. These results represent the flexibility and mobility chains of the polymer blend matrix. For all LDPE/m-PEwax/PLST blends containing 2 to 8 wt % of m-PEwax, except the blend with 10 wt % of m-PEwax, that showed immiscibility and phase separation, increasing the ratio of m-PEwax, which contains randomly distributed branch chains, increases the flexibility and elongation percentage. As a result, the blends that were compatibilized with 8 wt % of m-PEwax have mechanical properties approximately close to the mechanical properties of LDPE/PLST without compatibilizer. Similar behavior was found when PEwax filled LDPE. [17]

Rheological properties of blends

Capillary Rheometry is one of the commonly used methods to analyze the rheological properties of polymers. To demonstrate the rheological properties of polymers, melt rheology data calculated using a capillary rheometer can be recorded using various rheograms, i.e., shear stress vs. shear rate curve, viscosity vs. shear rate curve, and viscosity vs. temperature. Figures 3 and 4 highlight the results for the shear stress and the apparent viscosity as a function of the shear rate at 150°C (typical temperature for standard processing conditions), respectively. These figures showed that the curves deviate from a linear relationship and are inclined to the shear rate axis with an increasing shear rate. These results indicate that LDPE/PLST blends with m-PEwax are pseudoplastic non-Newtonian fluids. [29]

PE wax is usually used as a lubricating agent, [28] which can act as a plasticizer to promote melt slippage of blends on die wall. Therefore, it is reasonable to expect such shear stress reduction for LDPE/m-PEwax/PLST blend. The apparent viscosity of LDPE/PLST blends at various temperatures as a function of the wt % content of m-PEwax (Figure 5), revealed that in the addition of 2 wt % of m-PEwax, the viscosity of the blend decreased gradually with an increase in the m-PEwax weight ratio and it was decreased with the evaluated temperature at the same shear rate. PEwax is known for its low viscosity, lubricating property, and as processing aid. [22,28] In addition to glycerol, which is essentially a plasticizer for starch, m-PEwax may act as a minor plasticizer and facilitate melt slippage on a die wall by enhancing starch plasticization. As mentioned in the mechanical properties results, the addition of more than 2 wt % m-PEwax increased the flexibility of the chains in blends and decreasing the viscosity. However, the viscosity values remained lower than the viscosity values of blends without compatibilizer. Thus, as expected, m-PEwax decreased viscosity and increased the processability of the LDPE/m-PEwax/PLST blends. For LDPE/PLST blends using different weight percentages of m-PE wax as compatibilizer, a power-law equation was applied to determine the relationship between apparent shear stress and shear rate (flow curves). The equation for this model is given as follows:

$$\tau = K\gamma^n \ldots\ldots\ldots\ldots (4)$$

Where *n* is an index of power-law or an index of flow behavior, and *K* is an index of viscosity. From the linear relationship on the log-log scale, the slope equals *n* value, and the intercept equals *K* value.
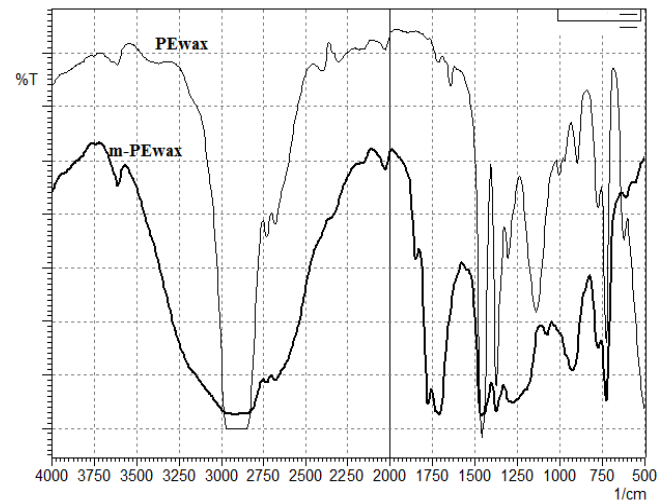
Figure 1: FTIR spectrum of polyethylene wax (PEwax) and maleated polyethylene wax (m-PEwax)
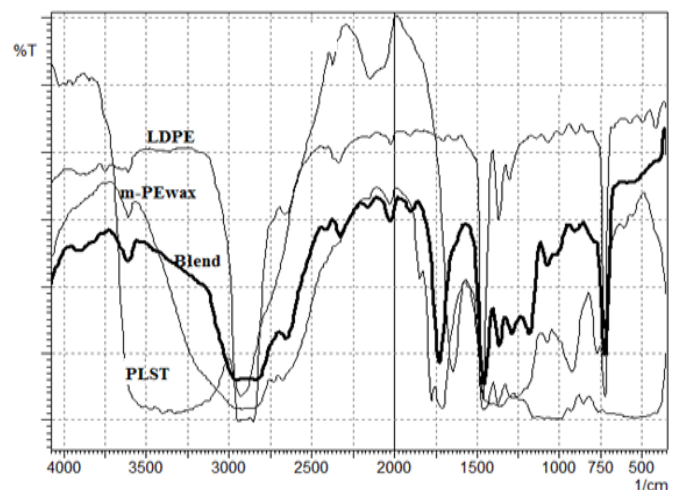
Figure 2: FTIR spectrum of low-density polyethylene (LDPE), plasticizer starch (PLST), maleated polyethylene wax (m-PEwax) and LDPE /m- PEwax/PLST blend (bold line)
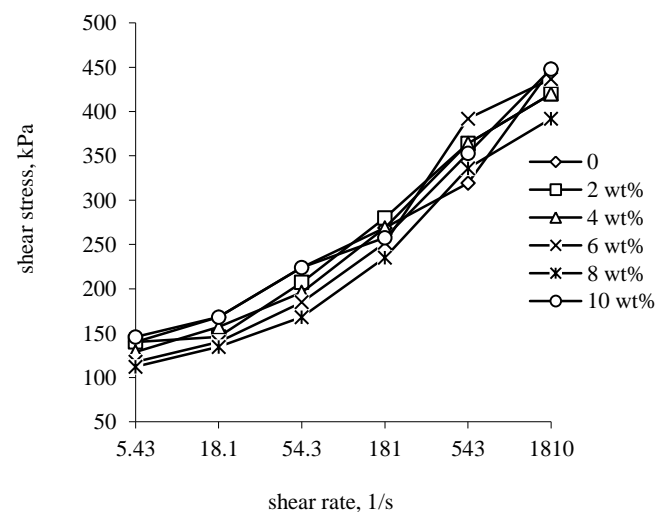
Figure 3: Shear stress vs shear rate for LDPE/PLST blends at 150°C

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The values of *n* and K of the LDPE/PLST blends at different temperatures are listed in Table 2. The slope of the linear equation was (1-*n*), and log K was the intercept. Increasing the m-PEwax ratio led to an increase in the flow index *n* and a decrease in K values. This result indicates that the addition of m-PEwax improved the sensitivity of the LDPE/PLST blends to the shear rate.

Alkyl groups and/or maleic anhydride homopolymer chains are among the chain branches found in m-PE wax. These chemical structures improved chain mobility of the LDPE/PLST blends when adding m-PEwax. Therefore, the n-value increased while the K value declined, except for blends containing 10 % m-PEwax, which had values similar to those of the mix without m-PEwax. The results indicate that the PLST phase was separated from the LDPE matrix, at 10 wt % of m-PEwax, due to poor interfacial adhesion between LDPE and PLST. A similar result was obtained from the above results of the mechanical properties according to the Arrhenius equation (5):

$$\eta = A.e^{Ea/RT} \ldots\ldots\ldots (5)$$

Where E_a is the viscous flow activation energy, A is the structure-related consistency and R is the gas constant (8.314 J mol^{-1} K^{-1}).

Table 3 shows the viscous flow activation energy E_a of the blends, which represents the temperature effect on blend behavior. High E_a values correspond to the high sensitivity of the blend to the temperature. The E_a values in Table 3 show that the LDPE/PLST blends with higher m-PEwax content were less responsive to processing temperatures. For m-PEwax content higher than 8 wt %, the blend shows a two-phase morphology, resulting in stress applied around the discrete domains. As a result, more energy is required to flow melt.
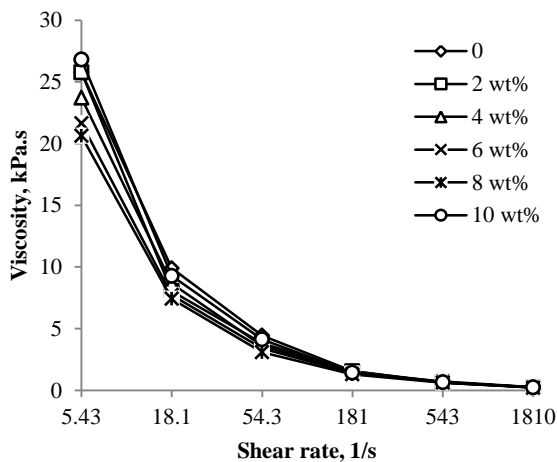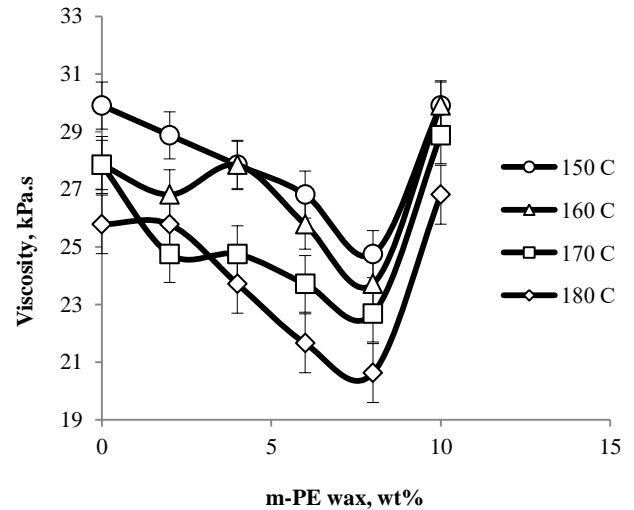

Figure 5: Viscosity vs m-PE wax content of LDPE/PLST at different temperatures

Table 1: Mechanical properties of LDPE/PLST blends with different weight percent of m-PE wax

% m-PE wax	Yield stress (MPa)	Break stress (MPa)	% Elongation at break	Break energy (J)
0	8.43	7.47	53.76	3.92
2	8.12	5.69	57.4	2.33
4	7.34	4.82	62.7	1.98
6	6.86	4.43	66.61	1.78
8	6.81	4.79	66.92	2.65
10	2.7	1.9	4.63	0.1

Figure 4: Viscosity vs shear rate for LDPE/PLST blends at 150 °C

Table 2: Flow behavior index (*n*) and consistency index (K) of LDPE/PLST blends at different ratios (wt %) of m-PE wax.

% m-PE wax	150 °C		160 °C		170 °C		180 °C	
	n	K	*n*	K	*n*	K	*n*	K
0%	0.0983	2.0403	0.0942	2.0787	0.0947	2.0933	0.0889	2.1323
2%	0.116	1.9096	0.1128	1.9522	0.1051	1.9826	0.1049	2.0497
4%	0.1236	1.9025	0.1151	1.9158	0.1094	1.9087	0.1056	2.0171
6%	0.168	1.8884	0.1366	1.881	0.1178	1.8648	0.1097	2.0054
8%	0.188	1.8052	0.1397	1.808	0.1235	1.8254	0.1091	2.0023
10%	0.0991	2.0446	0.0951	2.076	0.0854	2.1254	0.0838	2.1472

n: Index of flow behavior; K: Index of viscosity

Table 3: The activation energy (kJ/mol) of LDPE/PLST blends at different ratios (wt %) of m-PE wax

wt% m-PE wax	wt% m-PE wax		
	5.43	18.1	181
0 wt%	0.0193	0.0264	0.0203
2 wt%	0.0157	0.0227	0.0204
4 wt%	0.0114	0.0212	0.0183
6 wt%	0.0106	0.0211	0.0122
8 wt%	0.0102	0.0204	0.0097
10 wt%	0.0158	0.0395	0.0507

Conclusion

The findings from this study revealed that m-PEwax, as a compatibilizer, was partially compatible with components of the LDPE/PLST blends. The addition of m-PEwax to the LDPE/PLST blends reduced the mechanical performance of all the blends. Also, the rheological properties of m-PEwax blends were more sensitive to temperature than those of non-compatibilized blends.

Conflict of Interest
The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

Acknowledgments

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