Materials Physics and Mechanics 47 (2021) 103-116 Received: June 12, 2020

MELT RHEOLOGICAL BEHAVIOUR AND MECHANICAL PROPERTIES OF POLY(LACTIC ACID)/HIGH DENSITY POLYETHYLENE BLENDS

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Abstract. In this study, poly(lactic acid) was melt blended with high density polyethylene to investigate the rheological and mechanical properties of these blends. The rheological properties were characterized where viscous properties were evaluated in the molten state. In studying the viscous properties, non-Newtonian index (n), apparent viscosity, and the flow activation energy were determined. Mechanical properties were studied in terms of tensile properties, i.e. ultimate tensile strength, Young's modulus, elongation, and total energy at fracture. The results showed that PLA/HDPE blends and their pure components behave pseudoplastically ($n<1$), blends apparent viscosity decreases with increasing shear rate. It was also found that the curves of apparent viscosity versus blending ratio go through a minimum at blending ratio PLA/HDPE (75/25) vol% and that the PLA/HDPE (16/84) vol% blend has the lowest value of viscous activation energy. The mechanical results showed that the blends ultimate tensile strength and Young's modulus increased at up to 30 vol% PLA content. By contrast, the elongation and total energy at fracture versus blending ratio showed a continuous decrease up to PLA/HDPE (50/50) vol% ratio. These results comply with the behaviour of an incompatible polymer blend system.

Keywords: PLA, HDPE, polymer blends, viscosity, rheological properties

1. Introduction

One of the most important properties of plastic materials that contributed to its commercial success is its chemical inactivity and its resistance to degradation in addition to its low cost of production, light-weight, and compatibility with special applications [1,2]. The resistance of plastics to breakdown and degradation in different conditions is one of the important advantages of them during the investment, but this has led to the accumulation of plastic waste as a result of the excessive use of such materials and the emergence of a major problem for the environment that requires finding appropriate solutions [1,2]. In addition, most types of plastic materials used in daily life are derived from non-renewable sources such as crude oil and natural gas [1,2]. Among the solutions that have been proposed to solve these problems, is to recycle some types of plastic materials in order to be used again, but this method is not a final solution to the problem of plastic waste [1,2]. These problems led to an increased interest in developing polymers from renewable and environmentally friendly sources as an investment these types of polymers is one of the possible solutions in getting rid of the problem of growing plastic waste day after day and reducing dependence on plastic materials with oil sources [2]. Most Biodegradable polymers have high cost and low-performance properties and one of the options that available is blending biodegradable

http://dx.doi.org/10.18149/MPM.4712021_10

polymers with non-biodegradable polymers to obtain a biodegradable material with properties tailored to specific needs and it is the most versatile and economic method with a negotiation between cost and performance [3]. Poly Lactic Acid (PLA), as one of the most promising biodegradable thermoplastic polyester, has attracted attention not only in academia but also in the industry [4]. PLA is derived from renewable sources (corn, sugar beet) and it belongs to the family of linear esters derived from α-hydroxy acids and the main unit of the PLA is lactic acid [4]. PLA is used in many applications, it is used in drug delivery, tissue-engineering pharmaceutical, agricultural, and packaging applications [4,5]. PLA has mechanical properties that compete with petroleum-based polymers in use, for example the tensile strength value of the PLA is higher than that in HDPE, PP, and PS but the values of both impact and elongation at breakage are less than in these polymers [6]. High density polyethylene (HDPE) is a linear polyethylene prepared by polymerizing ethylene using a transition metal as Ziegler-Natta catalysts. HDPE has a low price and it is used in food and beverage packaging applications and drug packages in addition to many other applications [7]. PLA has some disadvantages such as brittleness, low thermal stability, and high price compared to PS, PP, and PE. There are many techniques used to overcome these disadvantages, and one of the most important of these techniques and the lowest cost is blending of polymers, where through the blending process can get material with improved properties. Also, blending a low-price polymer and low mechanical properties with another with a high price and good mechanical properties enables us to obtain a material with good properties at low cost, and in the case of PLA, it has been blended with many polymers, either biodegradable or non-biodegradable [8-11] to obtain new materials with low cost, improved properties, and biodegradability. There are many works that have studied the mechanical properties of PLA/HDPE blends. Lu et al. [12] have studied the mechanical properties of PLA/HDPE polymer blends. They found that their ultimate tensile strength increased with PLA content, but the elongation at break decreased. In the study of Machado et al. [13,14], mechanical properties of polyethylene and aliphatic polyesters blends were examined. It was found that the ultimate tensile strength and Young's modulus of the PLA/HDPE blends increased with increasing PLA content, whereas elongation at break decreased. Madhu et al. [15,16] prepared PLLA/HDPE blends and studied their mechanical properties. They found that the ultimate tensile strength and elongation at break of the blends decreased with PLA content. However, there is not much research has been done on studying the rheological properties of PLA/HDPE blends. This research will shed light on the concept of the mechanical properties of PLA/HDPE blends and focus on studying its rheological properties in the molten state.

2. Materials and methods

Polymers. Poly(lactic acid) (PLA) (ESUN™ A-1001) [density = 1.25 g/cm^3 (21.5°C), MFI = 12.5 g/10 min (190°C/2.16 Kg)] was supplied by Bright China Industrial Company. Ltd (Shenzhen, China), the selected grade is an extrusion material; it was dried at 70°C for 6 h before use. High density polyethylene (HDPE) (SABIC®HDPEM80064) [density = 0.964 g/cm^3, MFI = 8 g/10 min (190°C/2.16 Kg)] was supplied by Sabic (KSA).

Blends Preparation. The PLA/HDPE blends were mixed in the molten state using Brabender plastograph. The basic parameters of blending operation conditions are listed in Table 1. The compositions of the PLA/HDPE blends used in this work are shown in Table 2.

Table 1. Blending operation conditions

Blend	Blending time, min	Blending temperature,°C	Rotation speed, RPM
PLA/HDPE	15	190	30

Table 2. Composition of the PLA/HDPE blends

PLA/HDPE blends	PLA (wt%)	HDPE (wt%)
PLA0	0	100
PLA10	10	90
PLA20	20	80
PLA30	30	70
PLA40	40	60
PLA50	50	50
PLA60	60	40
PLA70	70	30
PLA80	80	20
PLA90	90	10
PLA100	100	0

Rheological characterization. Rheological properties of the blends were studied using capillary rheometer. The melt is extruded by the use of dead weights. The rheological experiments were carried out at 170°C, 180°C, and 190°C using L/R = 8, 15, 25, and 36 capillaries. Apparent shear rate (γ_a) was determined by the following equation [16]:

$$\gamma_a = \frac{4Q}{\pi R^3}, \tag{1}$$

where Q – volumetric flow (cm^3/s), R – radius of the used capillary die (cm).

$$\tau_a = \frac{P\,.\,R}{2\,.\,L}, \tag{2}$$

where P is the pressure at the capillary entrance (Pa), L is the capillary length (cm).

Apparent viscosity (η_a) is given by the following equation [16]:

$$\eta_a\,(\mathrm{Pa.s}) = \frac{\tau_a}{\gamma_a}\ . \tag{3}$$

The values of flow activation energy at constant shear stress (E_τ) were determined using Arrhenius equation form [17]:

$$\eta_a = A.\,e^{\frac{E_\tau}{R.T}}\ , \tag{4}$$

where η_a – apparent viscosity (Pa.s), R – gas constant (8.314 J/mol.K), T – absolute temperature (K), A – constant and E_τ – flow activation energy.

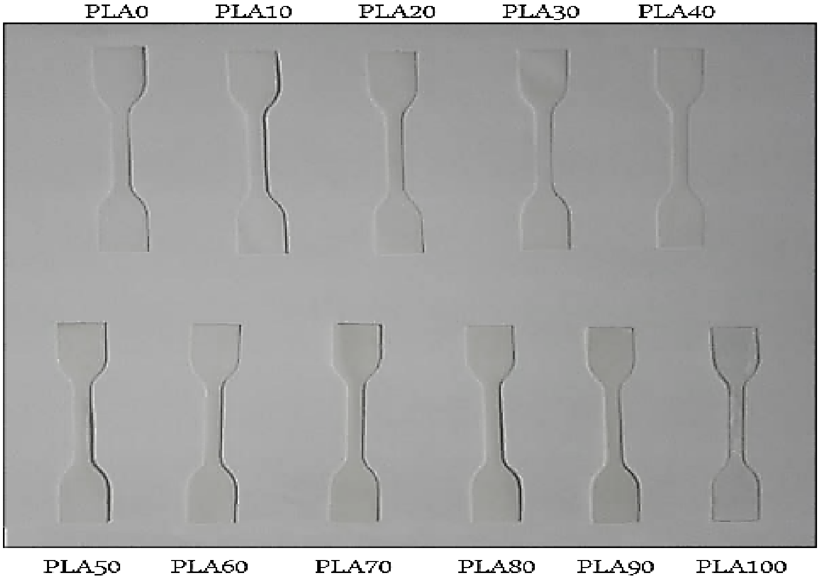

Fig 1. Tensile specimens of PLA/HDPE blends

Mechanical characterization. Tensile tests to determine ultimate tensile strength (MPa), Young's modulus (MPa), elongation (%), and energy (Joule) was performed using the universal mechanical testing machine (Servo hydraulic – Adamel Lhomargy DY-34, MTS, Roissy-en-Brie, France) at room temperature according to ASTM D 882. All specimens were subjected to tension at a displacement velocity of 5 mm/min. Results from 5-6 specimens were averaged. Figure 1 shows an arrangement of the specimens of PLA/HDPE blends.

3. Results and Discussion

Flow Curves. Flow curves (Figs. 2 and 3) show the variation of apparent shear stress (τ_a) versus apparent shear rate (γ_a) for PLA/HDPE blends at (L/R = 15, T = 180ºC).

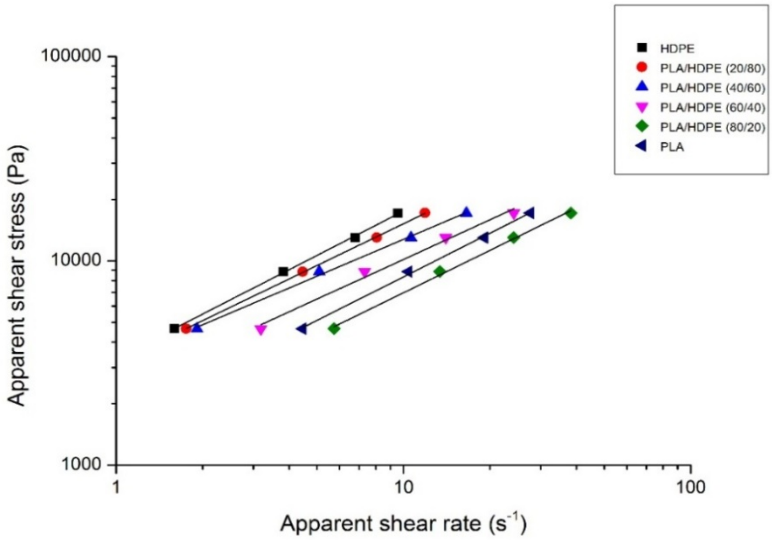

Fig. 2. Flow curves of (PLA0, PLA20, PLA40, PLA60, PLA80, PLA100) blends

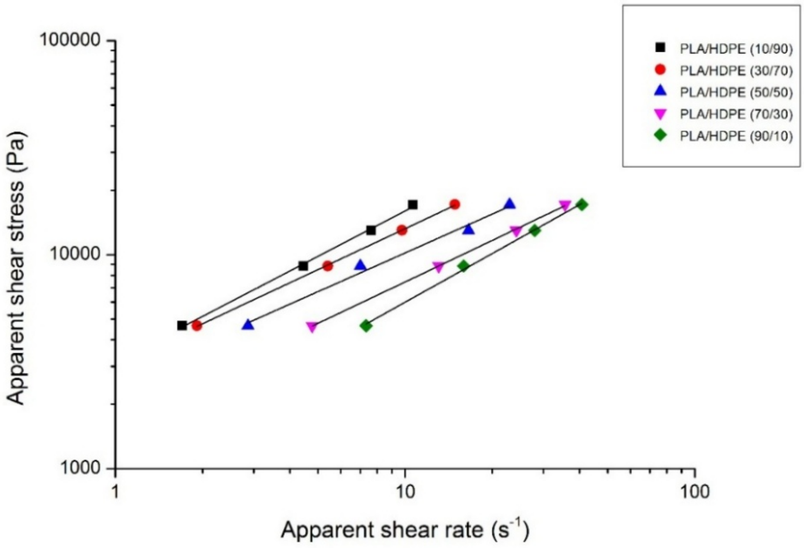

Fig. 3. Flow curves of (PLA10, PLA30, PLA50, PLA70, PLA90) blends

It can be seen that the curves of apparent shear stress (τ_a) versus apparent shear rate (γ_a) for PLA/HDPE blends are close to linearity. It means that the relation between (τ_a) and

(γ_a) for the PLA/HDPE blends accords with the power law, and the equation for this model is given as follows [17]:

$$\tau_a = K.\gamma_a^n, \tag{6}$$

where n is the flow index and K reflects the consistency index of the polymer melt.

n values were calculated from the slope of the fitted lines in Figs. 2 and 3 as stated in the following equation [17]:

$$n = \frac{d\log\tau_a}{d\log\gamma_a}\ . \tag{7}$$

The values of n are listed in Table 3.

Table 3. The values of n of PLA/HDPE blends

n	PLA%	0	10	20	30	40	50	60	70	80	90	100
	180°C	0.72	0.70	0.63	0.63	0.59	0.60	0.64	0.64	0.68	0.75	0.71

It can be seen that all n values were less than 1 for all studied blends. This indicates that PLA/HDPE blends in their rheological behaviour belong to pseudoplastic materials [18]. The flow index value indicates the extent of viscosity change by the shear rate at a given temperature and the changes of viscosity by shear rate changes are more when the value of n is less than one. Thus, at a temperature of 180°C, the viscosity of the PLA40 blend is less stable with respect to the shear rate change, while the PLA90 blend has a lower viscosity with respect to the shear rate change compared to the rest of the blends.

Melt Viscosity. Viscosity curves (Figs. 4 and 5) display the curves of apparent viscosity (η_a) versus apparent shear rate (γ_a) for PLA/HDPE blends at (L/R = 15, T = 180°C).

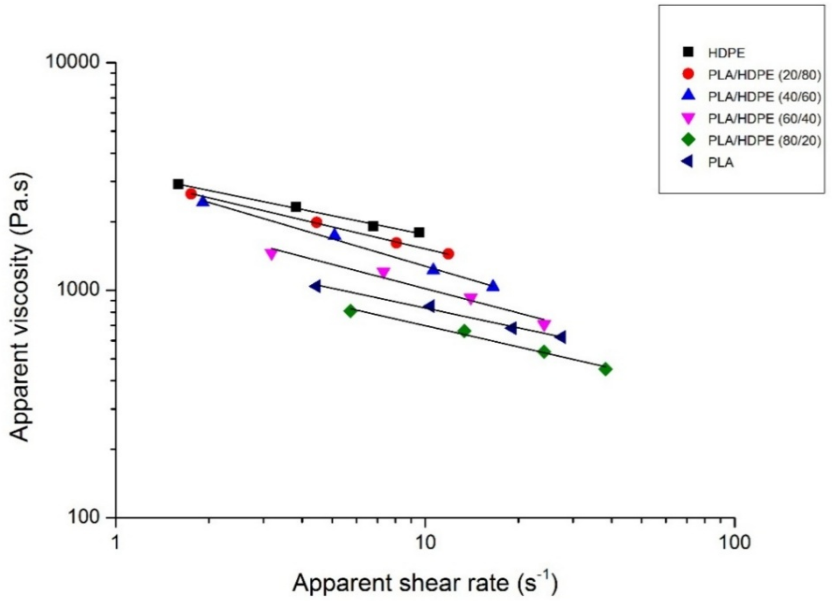

Fig. 4. Viscosity curves of (PLA0, PLA20, PLA40, PLA60, PLA80, PLA100) blends

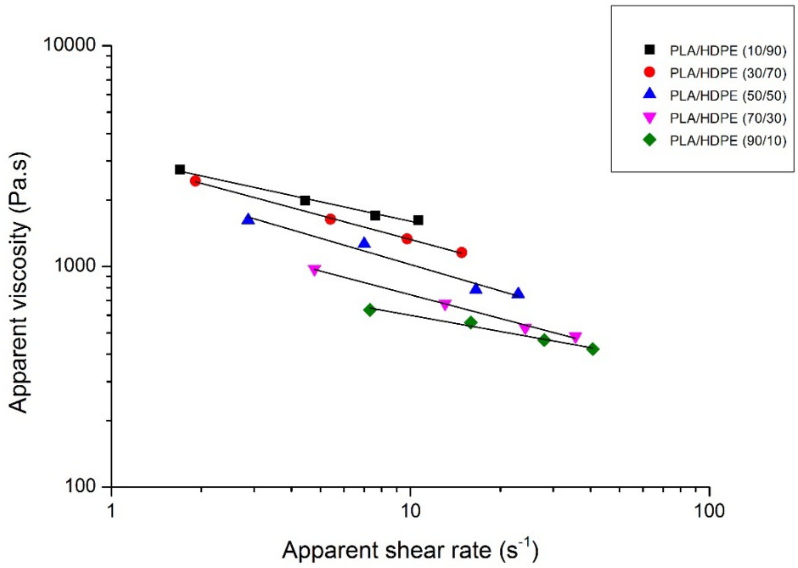

Fig 5. Viscosity curves of (PLA10, PLA30, PLA50, PLA70, PLA90) blends

It can be seen that for all PLA/HDPE blends, the viscosity decreases with increasing shear rate. Polymer chains are placed in the direction of the applied force, this leads to decreased viscosity [17]. The change of viscosity by changing the shear rate or shear stress plays an essential role in molding technologies of the materials, at the low shear rate corresponding to high viscosity, the molding process is achieved extrusion is best suited to keep the extruded material. Stable and homogeneous shape, while it is the injection molding process is best suited for low viscosities, which correspond to high shear rate, since low viscosity in the high field of shear rate leads to Reducing both the pressure and time required to fill the formation mold and thus a shorter injection cycle and at a lower cost of molding.

Effect of Composition on the apparent viscosity. Figure 6 displays the effect of the blending ratio on the apparent viscosity at constant shear stress.

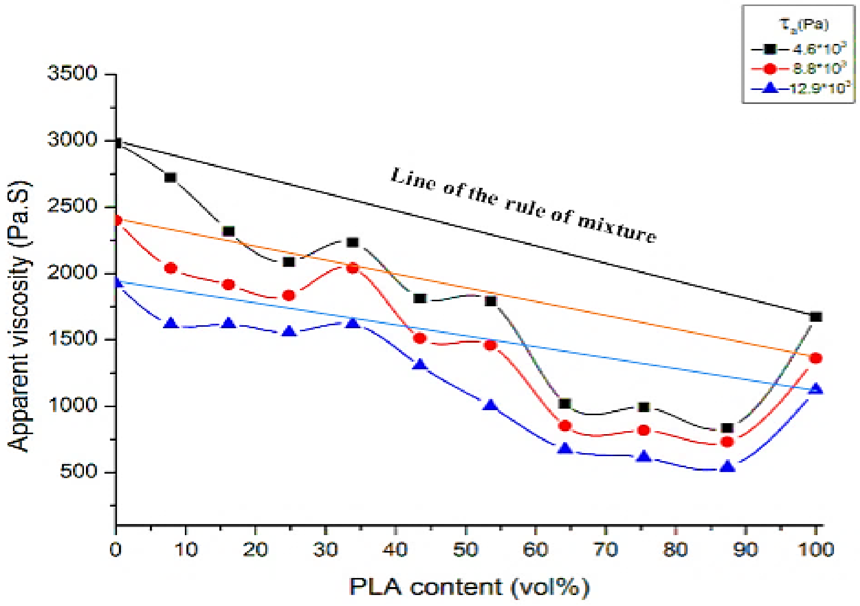

Fig. 6. Apparent viscosity of PLA/HDPE blends versus PLA content (vol%)

It can be clearly seen from Fig. 6, that the viscosity of HDPE is higher compared to that of pure PLA, the viscosity of blends vary between those of the pure polymers, and the viscosity of the blend decreases with increasing PLA content in the blend. This effect becomes more and more pronounced as PLA content increases. This behaviour can be attributed to the low melt viscosity of PLA compared with HDPE. These results are important since they indicate that the optimal processing conditions for shaping operations of PLA/HDPE blends could be quite different as compared to those for pure PLA. On the other side, the curves of apparent viscosity versus blending ratio go through a minimum value nearly at blending ratio PLA/HDPE (75/25) vol%. Therefore, it will be easier to process this blend composition. It can also be seen from Fig. 6 that the blends' apparent viscosity values being below those on the line of the rule of mixture, could be considered as an indication of typical immiscible polymer blends. So, this leads to the conclusion that PLA and HDPE are immiscible as the apparent viscosity values were far below the line of the rule of mixture (negative deviation) [18].

Flow Activation Energy. The effects of temperature on flow behaviour can be understood through the viscosity curves for the composite melts at different temperatures. Figures 7 and 8 display the relationship between apparent viscosity and 1/T of PLA/HDPE blends at constant shear stress.

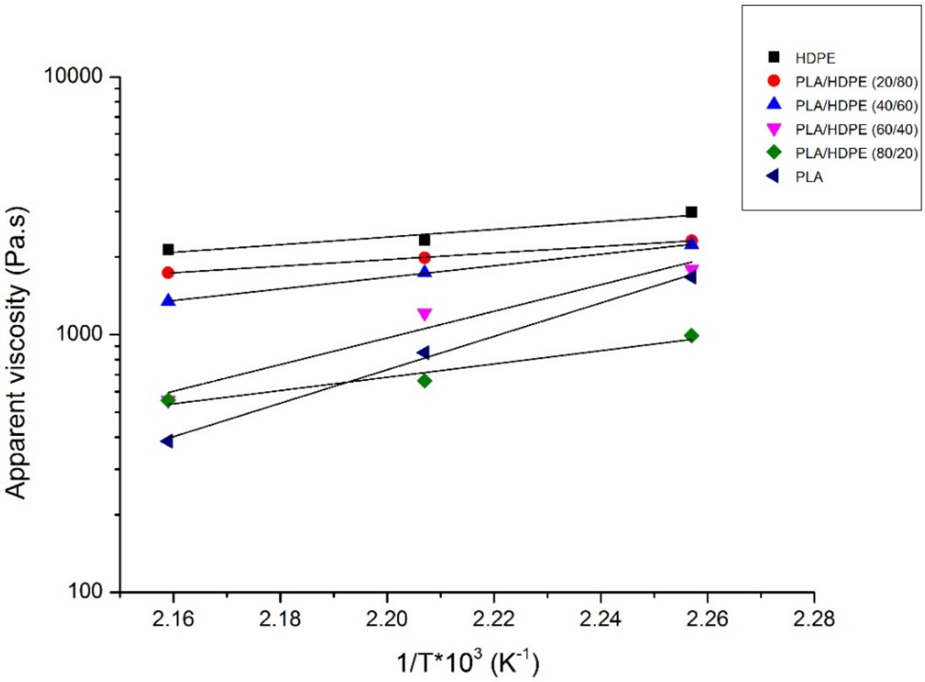

Fig. 7. Apparent viscosity versus 1/T of (PLA0, PLA20, PLA40, PLA60, PLA80, PLA100) blends

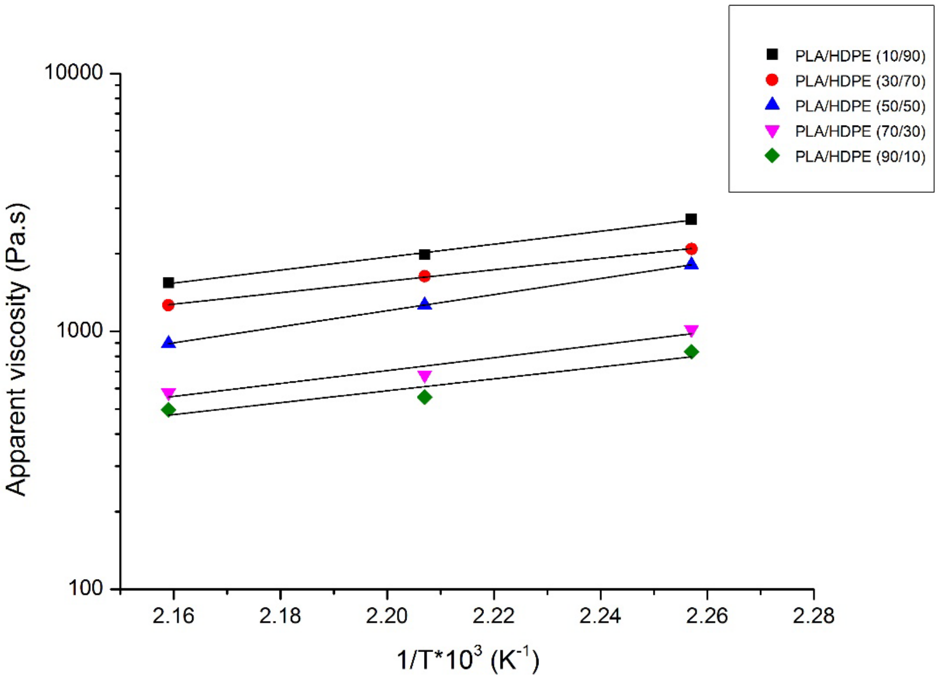

Fig. 8. Apparent viscosity versus 1/T of (PLA10, PLA30, PLA50, PLA70, PLA90) blends

It can be seen from Figs. 7 and 8 that the viscosity of PLA, HDPE, and all of their blends decreases with increasing temperature. However, the melt viscosity of PLA changes with temperature much more sharply than that of HDPE so, the flow behaviour of PLA is more sensitive to temperature than HDPE. The decrease in viscosity by increasing the temperature due to that when the temperature increases the melt free volume increases which reduction interactions and friction force between polymer chains, leading to a decrease in viscosity [19]. The temperature dependence of the viscosity of the blends can be characterized by the flow activation energy calculated from the Arrhenius equation as stated in the following equation [19]:

$$E_\tau = R\left(\frac{d\log\eta}{d\,1/T}\right), \tag{8}$$

where η is viscosity, E_τ is activation energy at constant shear stress, T is absolute temperature, and R is the universal gas constant.

Flow activation energy at constant shear stress (E_τ) can be calculated from the slopes of lines in Figs. 7 and 8. The values of (E_τ) are listed in Table 4 and Fig. 9 shows the effect of the blending ratio on the flow activation energy.

It can be seen from Fig. 9 that the flow activation energy of PLA is greater than that of HDPE which indicates that the flow behaviour of PLA is more sensitive to temperature compared with HDPE and the flow activation energies of PLA/HDPE blends are between those of PLA and HDPE. It could be noted from Table 3 that the viscosity of the blend PLA/HDPE (16/84) vol% which has the smallest value of (E_τ) at $\tau_a = 4.6\times10^3$ Pa and $\tau_a = 8.8\times10^3$ Pa will be stable over a wider range of temperature, at given shear stress whereas the blend PLA/HDPE (55/45) vol% has the greatest value of (E_τ) which indicates that the flow behaviour of this blend is most sensitive to temperature compared with other PLA/HDPE blends. The blends flow activation energy values are below the line of the rule of mixture, so it can be noticed that PLA and HDPE are immiscible.

Table 4. The values of flow activation energy of PLA/HDPE blends at constant shear stress

PLA/HDPE wt%	τ_a (Pa)	E_{τ_a} (kJ/mol)
0/100	4.6×10^3	10.74
	8.8×10^3	12.30
	12.9×10^3	11.17
10/90	4.6×10^3	8.21
	8.8×10^3	20.89
	12.9×10^3	11.07
20/80	4.6×10^3	6.94
	8.8×10^3	10.60
	12.9×10^3	13.78
30/70	4.6×10^3	15.83
	8.8×10^3	18.48
	12.9×10^3	19.69
40/60	4.6×10^3	13.08
	8.8×10^3	18.68
	12.9×10^3	21.76
50/50	4.6×10^3	25.59
	8.8×10^3	25.94
	12.9×10^3	27.02
60/40	4.6×10^3	47.19
	8.8×10^3	42.97
	12.9×10^3	43.75
70/30	4.6×10^3	26.20
	8.8×10^3	20.80
	12.9×10^3	24.83
80/20	4.6×10^3	25.56
	8.8×10^3	21.41
	12.9×10^3	34.60
90/10	4.6×10^3	20.65
	8.8×10^3	19.19
	12.9×10^3	33.75
100/0	4.6×10^3	55.90
	8.8×10^3	53.93
	12.9×10^3	48.54

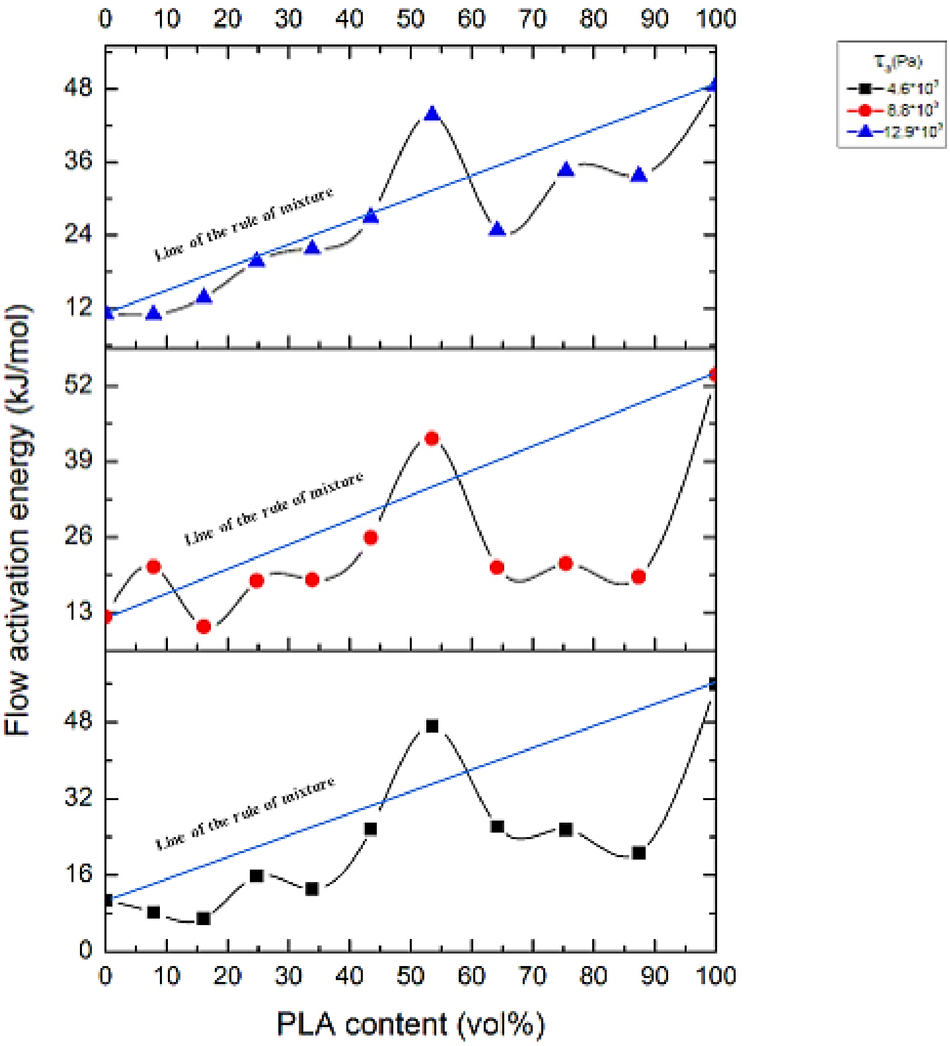

Fig. 9. Flow activation energy of PLA/HDPE blends versus PLA content (vol%)

Evaluation of mechanical properties. The results of the tensile properties of PLA/HDPE blends are given in Table 5.

Ultimate tensile strength of PLA/HDPE blends. Figure 10 shows the relationship between the ultimate tensile strength of PLA/HDPE blends and PLA content. It can be seen from Fig. 10, that the mechanical parameter of PLA/HDPE blends increases up to 30 vol% PLA content, shows a minimum at blending ratio PLA/HDPE (50/50), and becomes increasing at more than 50 vol% PLA content. In the first HDPE rich composition interval, the addition of PLA to HDPE contributes to HDPE reinforcement. By contrast, in the PLA rich composition interval, adding HDPE leads to PLA weakening. Within the intermediate composition interval, of PLA/HDPE blends, the mutual incompatibility of both PLA and HDPE results in a significant weakening of the blends ultimate tensile strength.

Table 5. Tensile properties of PLA/HDPE blends

PLA/HDPE blends	Ultimate tensile strength (MPa)	Young's modulus (MPa)	Elongation (%)	Total fracture energy (Joule)
PLA0	29.67 (±6.9)	889.78 (±126.35)	8.8 (±1.73)	3.61×10^{-2} $(\pm0.008)^{*}$
PLA10	31.25 (±1.62)	1082.86 (±55.86)	6.43 (±0.72)	2.79×10^{-2} (±0.0037)
PLA20	38.77 (±6.2)	1443.39 (±150.14)	5.16 (±0.5)	1.72×10^{-2} (±0.0032)
PLA30	39.29 (±4.82)	2039.64 (±175.89)	3.2 (±0.81)	0.74×10^{-2} (±0.0022)
PLA40	20.38 (±3.68)	1274.2 (±192.51)	2.56 (±0.45)	0.65×10^{-2} (±0.0028)
PLA50	15.97 (±1.92)	1129.98 (±109.23)	1.83 (±0.14)	0.49×10^{-2} (±0.00071)
PLA60	23.72 (±2.26)	1325.54 (±129.66)	2.15 (±0.18)	0.68×10^{-2} (±0.0018)
PLA70	26.64 (±2.85)	1430.07 (±139.48)	2.16 (±0.14)	1.02×10^{-2} (±0.0013)
PLA80	30.02 (±5.17)	1567.54 (±80.72)	2.23 (±0.31)	1.01×10^{-2} (±0.0027)
PLA90	38.36 (±3.24)	1639.89 (±95.94)	2.99 (±0.144)	2.05×10^{-2} (±0.0019)
PLA100	52.62 (±3.81)	1800.84 (±66.05)	3.86 (±0.16)	3.53×10^{-2} (±0.0057)

()*:Standard deviations

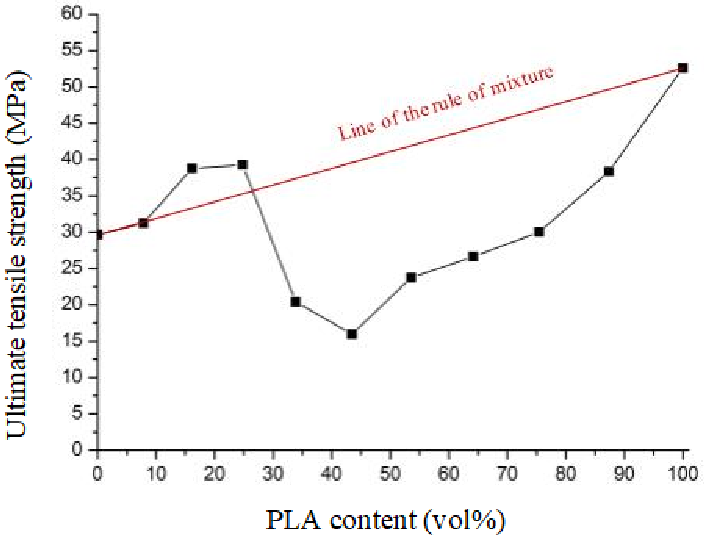

Fig. 10. Ultimate tensile strength of PLA/HDPE blends versus PLA content (vol%)

Young's modulus of PLA/HDPE blends. Young's modulus as a function of PLA content is shown in Fig. 11, Young's modulus shows two general stages. The first one follows the mechanical parameter increase up to the maximum value captured while the second one is located lower expressing its linear course at the PLA content of 40vol%÷100vol%. This result can be explained by the mechanical properties of the individual components (HDPE, PLA). Values of Young's modulus for PLA are higher than that for HDPE, so the addition of PLA to HDPE yields a material with a higher Young's modulus, and the best improvement in Young's modulus parameter was achieved at the PLA content of 30vol% which has the maximum value of Young's modulus.

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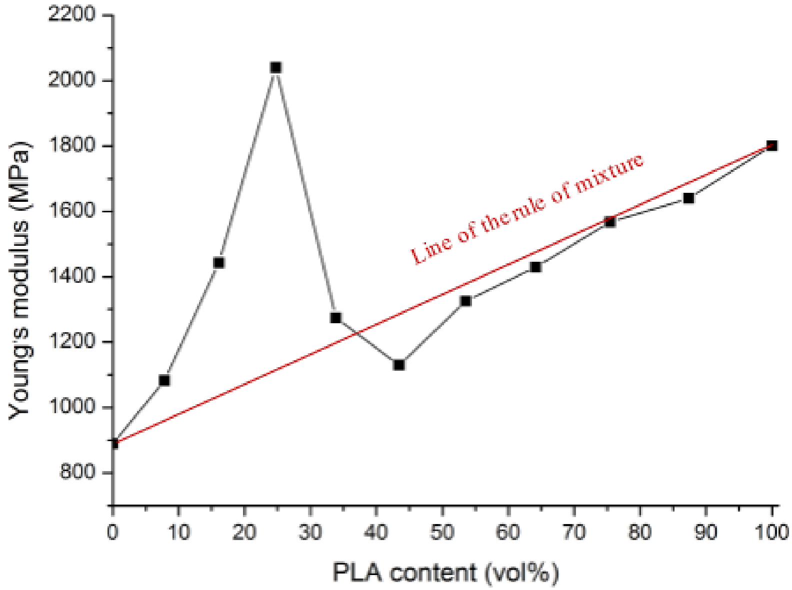

Fig. 11. Young's modulus of PLA/HDPE blends versus PLA content (vol%)

Elongation of PLA/HDPE blends. Figure 12 shows the relationship between elongation and PLA content for the PLA/HDPE blends. It can be seen from Fig. 12, that the elongation of PLA/HDPE blends decreases up to 50 vol% PLA content and becomes increasing at more than 50 vol% PLA content. The addition of PLA increases the rigidity of the blends, yielding materials with lower elongation.

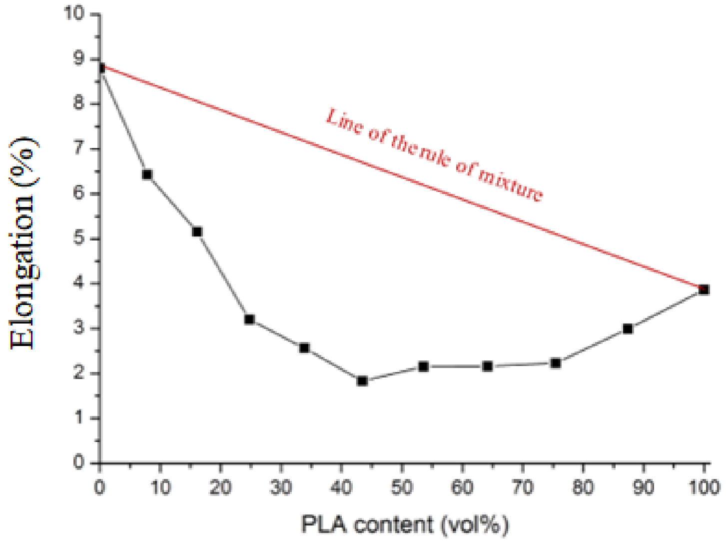

Fig. 12. Elongation of PLA/HDPE blends versus PLA content (vol%)

Total energy at fracture of PLA/HDPE blends. The total energy up to fracture as a function of PLA content is shown in Fig. 13, the parameter shows nearly a similar behaviour as elongation. The addition of PLA increases the rigidity of the blends, yielding a material with lower elongation and total energy at fracture.

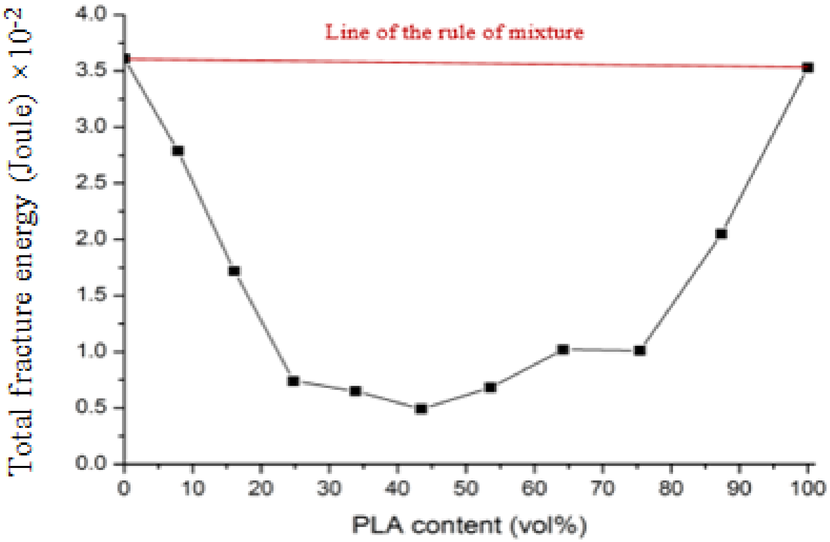

Fig. 13. Total energy at fracture of PLA/HDPE blends versus PLA content (vol%)

The blends mechanical properties values are below those on the line of the rule of mixture, so it was to say that PLA and HDPE form an immiscible polymer blend system [20].

4. Conclusion

In the present contribution, melt rheological behaviour and mechanical properties of PLA/HDPE blends were investigated. The relationship between apparent shear stress and the apparent shear rate was described by the power law. PLA/HDPE blends and their pure components behave pseudoplastically and the viscosity of the blend decreases with increasing PLA content. The dependence of viscosity on test temperature was studied, and the relationship obeys the Arrhenius expression. The flow activation energy of PLA is greater than that of HDPE and the flow activation energies of PLA/HDPE blends are between those of PLA and HDPE. The effect of the blend ratio on the mechanical properties of PLA/HDPE blends is investigated. The results showed that PLA addition enhanced both ultimate tensile strength and Young's modulus. By contrast, PLA addition lowered continuously up to 50 vol % both elongation and total energy at fracture of the blends. All these observations show that PLA and HDPE form an incompatible polymer blend system.

Acknowledgements. *The authors declare that there is no conflict of interests regarding the publication of this paper.*

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