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Thermal, mechanical and rheology of EVA/wax and wax/LLDPE blends as a carrier vehicle for investment casting pattern

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ABSTRACT

Different ratios of wax/ethylene vinyl acetate (EVA) and wax/linear low-density polyethylene (LLDPE) blends were prepared using one-step extrusion process to investigate their potential as carrier vehicles for pattern material for investment casting. The thermal, mechanical and rheological properties were characterised. Thermal analysis was done by thermogravimetric analysis (TGA) and differential scanning calorimetry. Mechanical properties were characterised by three-point bending and thermomechanical analysis, whereas the rheological properties were characterised by oscillatory rheometry. The TGA analysis showed that the incorporation of EVA or LLDPE into wax matrix improved the thermal stability properties of the blend. This can be attributed to an enhanced phase adhesion. The melting and solidification behaviour of the blends had intermediate temperatures between wax and EVA. The EVA/wax blends displayed evident viscosity shifts as compared to the viscosity of wax. The incorporation of EVA into wax significantly altered its mechanical properties. Fourier transform infrared spectrometry for both wax/EVA and wax/LLDPE showed a predominant presence of CH₂ and carbonyl group in the blend, and the mechanical properties of neat wax were improved when EVA and LLDPE were incorporated into wax.

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KEYWORDS

Wax/EVA; wax/LLDPE; rheology; thermal analysis; three-point bending

1. Introduction

Presently, there has been an immense concern in the development of wax blends or polymer blends with good thermal and mechanical stability for investment casting patterns [1]. With an increase in demand for tightly dimensional and complex shapes, various materials such as fillers, resins and plasticisers have been blended with wax to develop a more suitable pattern material for investment casting process. This has been done in recent years to overcome performance limitation exhibited by unfilled wax [2,3]. The identification of new materials to replace or improve the properties of conventional materials through blending technique has drawn lots of research and development of new polymer blends [4–6].

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Fischer–Tropsch waxes are synthetic waxes produced by gas–liquid technology by polymerisation of synthetic gas under elevated temperature and pressure into hydrocarbons [7,8]. Fischer–Tropsch waxes are characterised by low viscosity, high degree of linear molecular structure, high crystallinity and high congealing point [9]. Ethylene vinyl acetate (EVA) is a commercial copolymer consisting of ethylene and vinyl acetate (VA) units that has wide application such as a medium for filler dispersion and polymer compatibilization. The VA content in EVA varies from 3 to 50 weight percent [10,11].

Blends produced from wax and EVA have increased hardness with the copolymer concentration and decreases with decreasing VA content [12]. Thermal stability studies and the crystallisation behaviour of materials, polymers and polymer blends are valuable in the field of applied materials, science and technology [13]. In order to develop durable industrial products, it is necessary to investigate the thermal stability of these blends. Thermal analysis studies, such as investigation of the change in heat flow and stability of materials, will give some idea on the extent of chemical interaction occurring between the components, bond strength, activation energy, melting temperature and degradation kinetics [14].

The thermal stability of the material used for pattern material for investment casting process depends greatly on the addition and compatibility of the mixtures used to make the pattern. Thermal degradation characteristics of pattern material are analysed by thermogravimetry [15]. Thermogravimetric analysis epitomises the variation of sample mass with a change in temperature. The stages of thermal breakdown, thermal stability, degree of degradation, as well as its temperature threshold are well illustrated in thermogravimetric curves [16]. The melting, crystallisation and the miscibility of pattern material for investment is an important criterion used to characterise polymers [17].

Mechanical properties of industrial pattern waxes are crucial properties when describing pattern performances during moulding and when under temperature change. Pattern material with good mechanical properties should be easy to handle during investment casting. The estimation of shrinkage factors and pattern deformation tendencies gives a better understanding of investment casting pattern design. Pattern hardness, ultimate tensile strength and yield strength are determined by three-point bending test and hardness test [17].

Previous studies by Jaw et al. [18] and Krupa et al. [19,20] investigated the blending wax with other polymeric compounds. Most of the previous studies focused on the thermal, mechanical and surface characterisation and the application of the wax blends as a hot melt's material. There is little or unknown study on the effects of blending paraffin wax on the penetration and viscosity studies [21].

The objective of this study was to investigate thermal, mechanical and rheological properties of EVA and LLDPE incorporated into wax matrix. The varied proportions of the EVA/wax and LLDPE/wax blends were prepared using a twin co-rotating twin screw extruder.

2. Materials and methods

2.1. Materials

Sasol Fischer–Tropsch wax (melting point = 55°C, average molecular weight = 785 g/mol, density at 25°C = 0.9 g/cm³, soft, brittle straight hydrocarbon wax) was supplied by Sasol

SA. Elvax 250 (28% vinyl acetate – melting point = 71°C and density at 25°C = 0.926 g/cm³, melt flow index (MFI) = 25) was supplied by Carst & walker SA, and LLDPE (density at 25°C = 0.93 g/cm³, MFI = 50) was supplied by Sabric, SA. The compounds initially prior to development were tested as received. The gases used in the thermal characterisation techniques of the pattern material were of high purity (99.99%) and of analytical grade sourced from Air Liquid SA and Afrox SA.

2.2. Method

The formulations were done according to Table 1. The formulations were compounded on a T×28P 28 mm co-rotating twin-screw extruder with an L/D ratio of 18. The screw design comprised intermeshing kneader elements with a forward transport action. The processing temperature profile, from hopper to die, was 40/80/90/100°C.

2.3. Characterisation and testing methods

2.3.1. Thermogravimetric analysis (TGA)

The thermal characterisation studies on the wax/EVA and wax/LLDPE blends were performed by TGA analysis and at a constant heating rate using Advanced Laboratories Solutions SDT Q 600 TGA analyser. A mass of 5–10 mg was degraded in a 70 µL alumina pan under nitrogen at a flow rate of 50 ml/min in the thermo balance under dynamic conditions at a heating rate of 20°C/min. The EVA and LLDPE/wax blends were scanned from 30°C to 900°C. Multiple experimental runs were performed to obtain consistent data.

2.3.2. Differential scanning calorimetry (DSC)

DSC analysis of the EVA/wax and LLDPE/wax blends was performed using Perkin Elmer DSC 4000 analyser under a constant dry nitrogen purge introduced into the DSC cell at 50 mL/min. The samples mass used was between 10 and 20 mg. The samples were weighed and filled into an aluminium pan. An aluminium lid was then sealed hermetically onto the pan. All analyses were performed in the scanning mode from 20°C to 200°C at a heating rate of 10°C/min.

2.3.3. Fourier transform infrared (FT-IR)

FT-IR spectroscopy of the samples was performed in transmission mode detection. The EVA/wax and LLDPE/wax blends were analysed using Perkin Elmer spectrum 100 FT-IR

Table 1. Experimental design for the wax/EVA and wax LLDPE blend formulations.

Wax (g)	EVA (g)	Wax (%)	EVA (%)	Wax (g)	LLDPE (g)	Wax (%)	LLDPE (%)
100	0	100	0	100	0	100	0
30	20	60	40	30	20	60	40
30	30	50	50	30	30	50	50
40	10	80	20	40	10	80	20
40	20	67	33	40	20	67	33
40	30	57	43	40	30	57	43
50	10	83	17	50	10	83	17
50	20	71	29	50	20	71	29

spectrometer from 4000 to 550 cm^{-1} at a resolution of 4 cm^{-1} spectra representing an average of 16 scans. About 1 mg of each EVA/wax and LLDPE/wax blends was placed in the accessory as the spectra was recorded.

2.3.4. Mechanical testing

Mechanical testing of the blends was performed by three-point flexural test according to ASTM 790 using an Instron 5564 twin column tensile tester with a 5 kN load cell. Rectangular specimens with dimensions of 10 × 10 × 80 mm were obtained by casting in an Amsil 25 cure silicone mould. At least five samples were tested for each specimen at a crosshead speed of 1.8 mm/min and the load applied over a 64 mm support span.

2.3.5. Thermomechanical analysis (TMA)

TMA of the blends was carried out using Advanced Laboratories TMA Q 400 analyser according to ASTM D696. The analysis was done at a force of 0.02–0.1 N, and the measurements were conducted at 30°C. TMA probe was fitted with a needle with a tip radius of 1.4 mm under nitrogen gas at 50 ml/min. The penetration measurement was obtained on the immediate direction of compacts.

2.3.6. Rheology

Viscosity was determined using Anton Paar MCR301 rheometer using the parallel plate configuration with a 25 mm ϕ spindle. The temperature was controlled at 90°C and 125°C. All the measurements were done under a nitrogen atmosphere in order to avoid thermal oxidation.

3. Results and discussion

3.1. TGA

Figure 1(a,b) shows the TGA curves of EVA/wax and LLDPE/wax blends. From Figure 1(a,b), it can be seen that both EVA and LLDPE exhibit a higher thermal stability as compared to neat wax. This can likely be attributed to the higher molecular weight of EVA and LLDPE than neat wax. As it can be seen from Figure 1(a), wax decomposes continuously from a temperature of 220°C to around 400°C. This can be attributed to the differing wax chains caused by the wax molecules breaking down into smaller hydrocarbons compounds with different molecular masses during thermal decomposition. The mass loss of the wax is most likely dominated by concurrent evaporation and thermal degradation events. EVA has high thermal stability as compared to wax with two mass loss peaks. The first mass loss peak at a temperature of around 345°C could be due to the decomposition of acetic acid, hence the loss of VA units via a deacylation process and another mass loss peak at around 450°C. The second mass loss peak can be attributed to the loss of methylenic chain resulting in the formation of double bonds of partially unsaturated polyethylene material polymer [22,23]. From Figure 1(b), it is also evident that LLDPE has one mass loss peak, and the mass loss can be ascribed to the degradation of the long chains. Also, from Figure 1(a,b), it is also evident that the different EVA/wax and LLDPE/wax blends have intermediate thermal stabilities with two mass loss

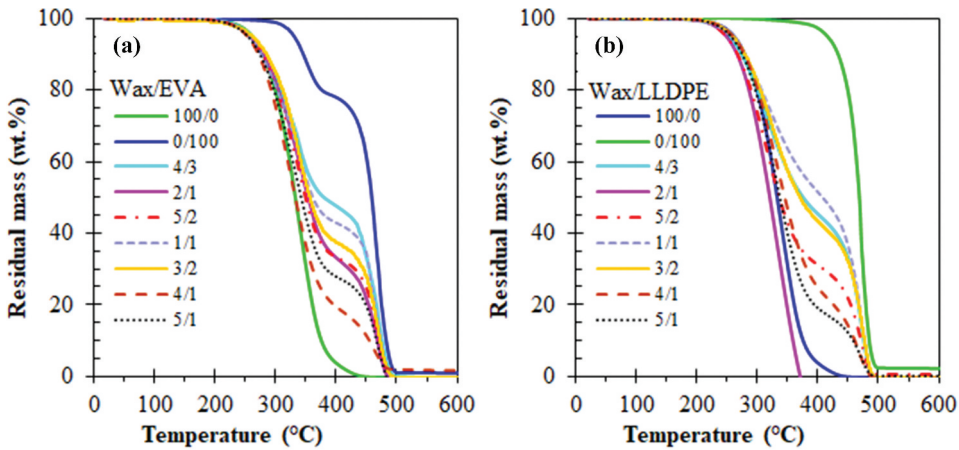


Figure 1. TGA mass loss curve of (a) EVA and EVA/wax blends and (b) LLDPE and LLDPE/wax blends.

distinct peaks at 265°C and 342°C for EVA/wax blends and 245°C and 365°C for LLDPE/wax blends. The first mass loss could be due to the evaporation of wax and the second mass loss peak can be ascribed to the degradation of EVA and LLDPE in the case of 1 (b). EVA/wax and LLDPE/wax binary blends decompose fully at temperatures above 480°C. The interaction between the wax and EVA and LLDPE and wax could be associated with the tie molecules of CH_2 chain interaction and better interfacial adhesion caused by interaction of the CH_2 chains, hence creating a better dispersion of wax, EVA and LLDPE into the matrices of the formed polymer blends.

3.2. DSC

Figure 2(a,b) show the DSC curves for neat EVA, wax, LLDPE, wax/EVA and wax/LLDPE blends during heating and Figure 3(a,b) during cooling. Heating was done to 150°C and cooled to 30°C and reheated to 150°C. From Figure 2, it can be seen that thermal profile curves for wax have two distinct endothermic peaks, whereas LLDPE has one distinct peak. The wax is completely molten above 70°C. The lower temperature peaks are due to a solid–solid phase transformation. The other peaks are due to the solid–liquid melting or crystallisation transitions. It can also be observed that the solid–solid transition peak of wax/EVA decreases in magnitude as the EVA content increases. This was also observed in the case of wax/LLDPE binary blends. In the case of wax/EVA blends, the separate peaks of wax were not observed since similar melting characteristics was witnessed due to wax and EVA melting in the same temperature range. From Figure 2(b), wax/LLDPE has two distinct peaks associated with the melting of wax and LLDPE. LLDPE and wax are not miscible with each other in the crystalline phase [18,19]. Generally, the lower temperature peaks are due to a solid–solid phase transformation. The other peaks are due to the solid–liquid melting or crystallisation transitions. During cooling, as shown in Figure 3(b), wax, EVA and wax/EVA binary mixtures showed two exothermic peaks. Figure 3(a,b) shows the cooling of wax, LLDPE, wax/EVA and wax/

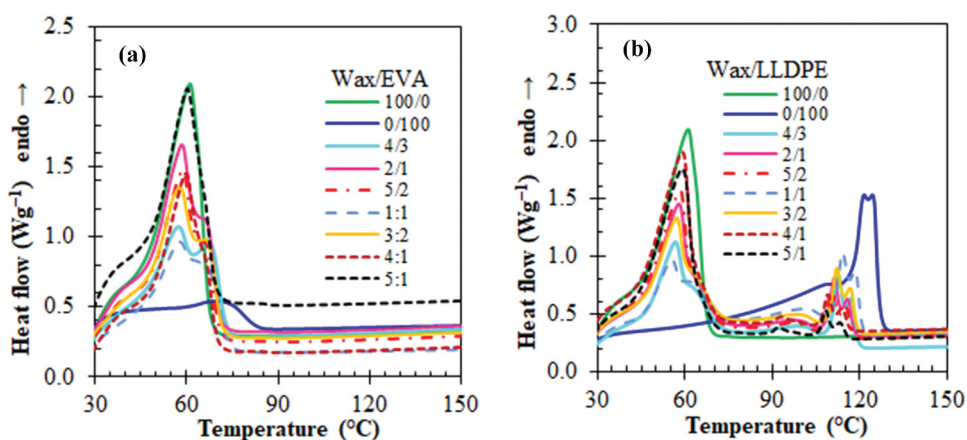


Figure 2. (a) DSC heating curves for wax, EVA and EVA/wax blends. (b) DSC heating curves for wax, LLDPE and wax/LLDPE blends.

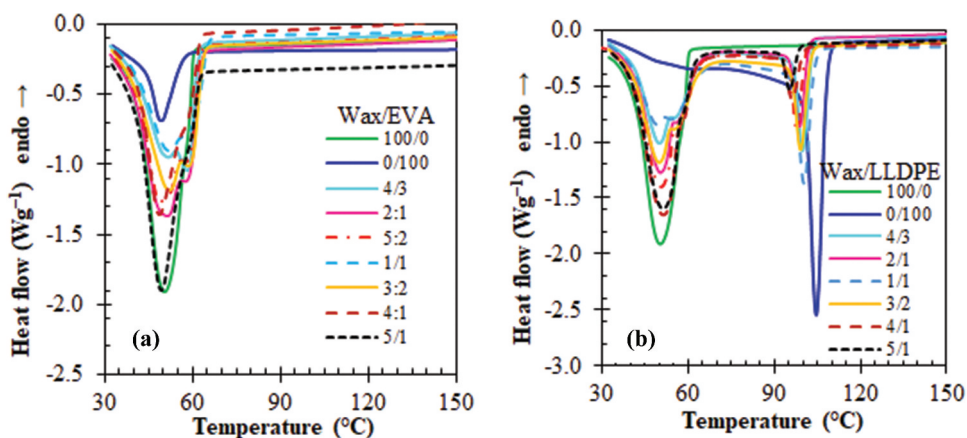


Figure 3. (a) DSC cooling curves for wax, EVA and EVA/wax blends. (b) DSC cooling curves for wax, LLDPE and wax/LLDPE blends.

LLDPE blends. From Figure 3(a,b), it can be concluded that at a higher wax content, there is a possibility of the larger part of wax not co-crystallising with both EVA and LLDPE; hence, wax will crystallise in LLDPE amorphous phase.

3.3. FT-IR

Figure 4(a,b) shows the FT-IR spectra of wax/EVA blends and wax LLDPE blends, respectively. Wax/EVA blends have five main bands in the wavelength range of 4000–500 cm⁻¹, whereas wax/LLDPE has three main peaks. Both EVA and LLDPE blends have a wide band in the region of 2919–2842 cm⁻¹ and in the region of 1459–1480 cm⁻¹. This can be attributed to the deformation vibrations stretch of a long-chain aliphatic hydrocarbon denoted by (C–H) and –C–H– bend of the alkane’s hydrocarbons, respectively. The less sharp bend indicated by both wax/EVA and wax/LLDPE at a band of between

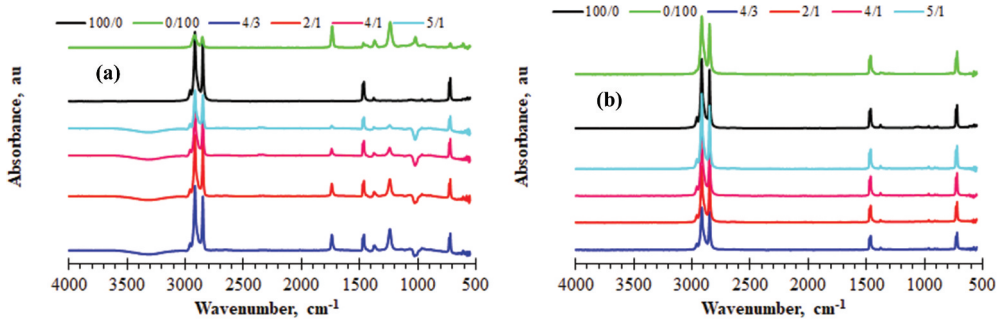


Figure 4. FT-IR spectra of (a) wax/EVA blends and (b) wax/LLDPE blends.

700 cm^{-1} and 634 cm^{-1} could be ascribed to $-\text{C}-\text{H}-$ bend of the alkane’s hydrocarbons. The wax/EVA spectra further showed a sharper peak at a band in the region of 1760–1665 cm^{-1} . This peak can be due to the presence of carbonyl absorption group denoted by $-\text{C}=\text{O}-$ stretch.

3.4. Three-point bending test

Figure 5(a,b) and Table 2 show typical three-point bending results of wax/EVA and wax/LLDPE binary blends. From Figure 5(a,b), it can be seen that an increase in EVA and LLDPE content in the binary blends leads to a marginal increase in bending strength. This can be attributed to EVA and LLDPE same CH_2 chains being interlinked with each other in the blends. The neat wax is very brittle and most blended samples showed similar brittle failure behaviour at lower wax percent with an abrupt fracture observed when the maximum load is reached. This can be due to an increase in the resistance to crack propagation. Generally, wax experiences a brittle failure at small flexural strains due to easy propagation of cracks through its crystalline structure. Table 2 reports on the mechanical properties of the wax and the formulated blends. Further, there is significant

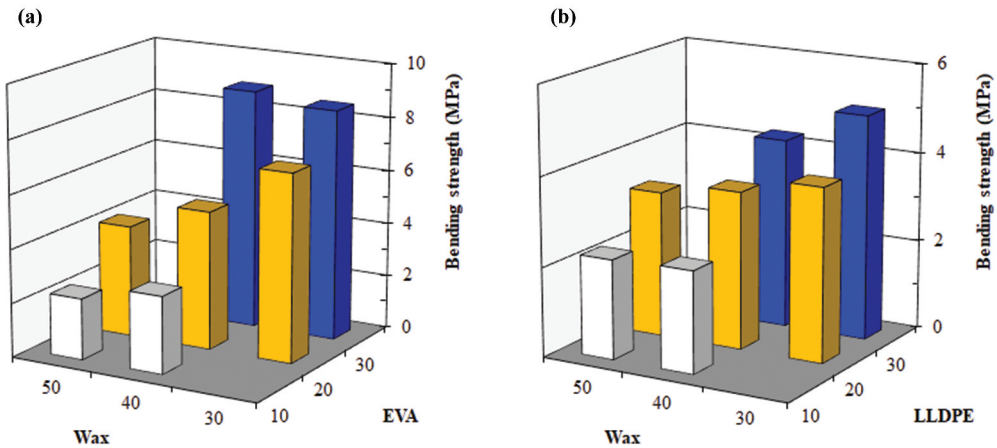


Figure 5. (a) Bending strength of wax/EVA blends. (b) Bending strength of wax/LLDPE blends.

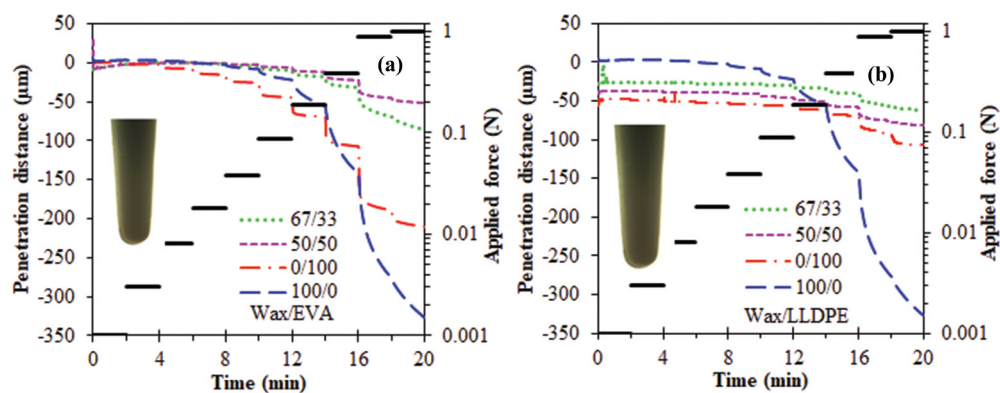
Table 2. Mechanical properties of wax/EVA and wax/LLDPE blends obtained from bending measurements.

Wax (g)	EVA (g)	Bending stress (MPa)	Modulus (MPa)	Wax (g)	LLDPE (g)	Bending stress (MPa)	Modulus (MPa)
100	0	1.3 ± 0.1	38 ± 20	100	0	1.3 ± 0.1	38 ± 20
30	20	7.0 ± 0.5	179 ± 21	30	20	3.9 ± 1.0	384 ± 123
30	30	5 ± 0.4	179 ± 19	30	30	5.0 ± 1.1	493 ± 196
40	10	2.9 ± 0.1	138 ± 12	40	10	2.3 ± 0.2	266 ± 68
40	20	5.1 ± 0.5	160 ± 28	40	20	3.8 ± 0.4	318 ± 35
40	30	8.9 ± 0.3	201 ± 11	40	30	4.3 ± 0.6	477 ± 145
50	10	2.3 ± 0.3	159 ± 29	50	10	2.2 ± 0.4	302 ± 55
50	20	4.1 ± 0.2	238 ± 88	50	20	3.3 ± 0.6	309 ± 41

influence of wax content on flexural modulus. This can be associated with the co-crystallisation of wax EVA and LLDPE. The addition of EVA and LLDPE into wax matrix clearly improves the bending strength in addition to increasing the deformation at break. In the case of flexural modulus as it can be seen in Table 2, as the wax content increases, there was an increase in the flexural modulus. Flexural modulus is dependent on the interaction between the crystalline and the amorphous regions of the blends due to elongation energy transmitted from the amorphous to the crystalline phase. Hence, as the wax content increases the crystallinity increases and the flexural modulus is expected to increase. In the case of LLDPE, the polyethylene chains have an effect on the energy transfer to the crystalline phase, hence the increase in the stiffness as compared to wax/EVA-based blends.

3.5. TMA

Figure 6(a,b) shows the TMA penetration curves of neat wax, wax/EVA and wax/LLDPE binary blends. As it can be seen in both Figure 6(a,b), as low force was applied, the needle penetrated slowly in all the blends. It can also be seen that neat wax is softer than EVA and LLDPE. The rate of penetration in all the binary blends decreased when force is applied during the full 2-min testing interval for all the blends. In both the EVA-based and LLDPE-based wax blends, the hardness of the binary blends formulated was higher

**Figure 6.** TMA curves of (a) wax and wax/EVA and (b) wax and wax/LLDPE. The inset shows the needle profile.

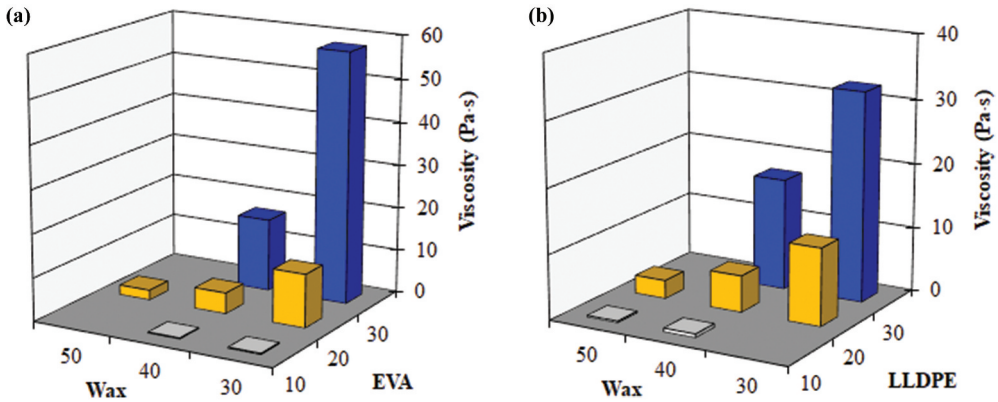


Figure 7. Viscosity of (a) wax/EVA and (b) wax/LLDPE blends measured at 90°C.

than that of wax, EVA and LLDPE. EVA and LLDPE forms a polymer blend with improved mechanical properties, though this is all dependent on the interaction between wax and EVA/LLDPE polymers ratios.

3.6. Rheology of wax/EVA blends and wax/LLDPE blends

The melt flow behaviour of wax/EVA blends and wax/LLDPE blends at a temperature of 90°C and at 125°C was measured as a function of shear rate. From Figure 7(a), it can be seen that the melt viscosity increased as the EVA and LLDPE content is increased, and EVA-wax-based blend had a slightly higher viscosity measurements than LLDPE-wax-based blends.

The viscosity of the blends follows a form of the composition dependence proposed by Arrhenius (Arrhenius, 1887) as modified by (Grunberg and Nissan, 1949) (Equation 4):

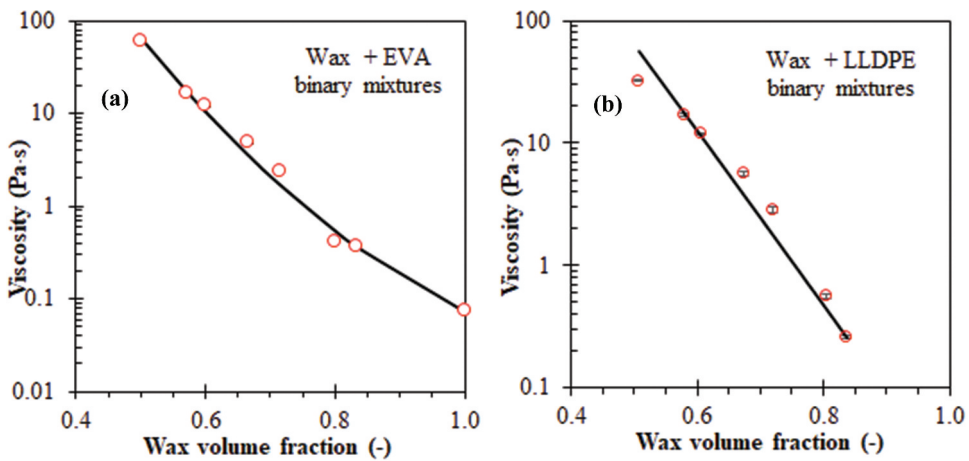


Figure 8. Viscosity of (a) EVA/wax blends and (b) effect of mass fraction of viscosity.

$$\eta_{\text{mix}} = \exp(w_{\text{wax}} \ln \eta_{\text{wax}} + 2w_{\text{wax}} w_{\text{EVA}} \ln \eta_{\text{Int}} + w_{\text{EVA}} \ln \eta_{\text{EVA}})$$

where η_{mix} is the viscosity of the binary mixture, and w_{wax} and $w_{\text{EVA/LLDPE}}$ are the mass fractions of the wax and EVA, respectively. The parameters η_{wax} and $\eta_{\text{EVA/LLDPE}}$ represent the viscosities of the neat wax and EVA, respectively.

The η_{Int} is an interaction term. This and $\eta_{\text{EVA/LLDPE}}$ were treated as adjustable parameters as it was not possible to accurately measure the viscosity of the EVA and LLDPE with the equipment available as they are not sufficiently fluid at 90°C. Least squares regression yielded and $\ln \eta_{\text{Int}} = -5.944$ and $\ln \eta_{\text{EVA}} = 16.98$ on setting $\ln \eta_{\text{wax}} = -2.615$. The solid line in [Figure 8\(a,b\)](#) shows the trend predicted by equation (4) with these values for the parameters.

4. Conclusions

The following conclusions were made:

- (i) The thermal stability of wax improved when both EVA and LLDPE were incorporated into the wax matrix. EVA content in wax/EVA binary blends causes an increase in the binary blend melting point during heating, whereas on cooling, the low crystallinity in EVA-28 means the congealing point is almost identical to that of paraffin wax. In the case of wax/LLDPE, crystallisation of the blend of LLDPE is strongly affected by the wax loading. Wax acts as a solvent for LLDPE; hence, its melting and crystallisation depends on the amount of wax and LLDPE.
- (ii) An increase in the EVA weight in the binary blend leads to retardation of the brittle fracture behaviour of the blend which results in the produced blend material having an increasing degree of plastic deformation before fracture. In the case of wax/LLDPE blends, the Young modulus, stress and strain increased with an increase in the amount of LLDPE. This could be attributed to higher degree of crystallinity experienced.
- (iii) The viscosity of neat paraffin wax increased when it was blended with EVA and LLDPE. At EVA and LLDPE weight loading of 30%, the viscosity was in the magnitude of almost 100 for LLDPE and almost 200 in the case of EVA. Both EVA and LLDPE had low MFI; hence, at higher weight ratios in the blends, the viscosities will increase by a higher margin. The Krieger Dougherty function (K-D) revealed that the experimental results in both EVA-based and LLDPE-wax-based blends agree with the correlation for the volume of fractions tested.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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