

Characteristics and Mechanical Properties Changes Due to Incorporation of Aloe Vera in Polyethylene-Based Film

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ABSTRACT

Non-degradable properties of polyethylene (PE) films due to long-chain structure cause increment of solid waste plastic. Many researchers, with different purposes, have studied the incorporation of functional materials to PE. Exploring the impact of the incorporation of aloe Vera (AV) into PE films in terms of its characteristic and mechanical properties is the main objective of this paper. The films were prepared using melt-blending and hot press technique. The characterisation assessed for the PE and PE-AV films were spectroscopy, crystalline phase, thermal analysis, and performance of the mechanical properties of the sample. Some of the AV functional groups disappeared upon addition with PE but has the possibility of two sharing peaks between AV and PE, which was 1472 and 2915 cm^{-1} . Lower thermal degradation temperature (T_d) obtained for PE-AV3 while other films found no significant changes in T_d value, and only one peak of thermal degradation occurred for all films. The same goes for the analysis obtained from differential scanning calorimetry (DSC) data. However, the crystalline structure displayed momentous peak changes for PE with AV. The highest tensile strength (TS) obtained by PE-AV3, at once, developing the highest value of Young's modulus (YM), modulus of resilience (U_E), and modulus of toughness (U_T). A certain amount of AV has a substantial effect on changing the polymeric structure, especially improving the mechanical properties of PE film. Therefore, AV has the potential to become an additive for developing a new, partially degradable PE film.



Keywords: *polyethylene, aloe vera gel, film packaging*

INTRODUCTION

Polyethylene is a type of synthetic polymer that has an extended chain ethylene monomer. PE has been used in many industries such as packaging, agriculture, automotive, constructions, consumer good, cosmetics, pharmaceuticals, and others. The packaging industry covers almost 50 % demand for PE in the year 2018 and expected increasing demand until the year 2026 [1]. Low cost, light, good chemical resistance, excellent processability, and raised in the mechanical properties are among the benefits of PE as an active film packaging (AFP) [2]. However, PE film cannot inhibit the bacterial growth on food and also causes environmental issues by increasing the amount of plastic waste disposal in the landfill due to the long-chain structure (non-degradable properties). Therefore, blending PE with antimicrobial agents can improve packaging ability. The additives can convert non-degradable PE film into a partially degradable film.

Several works reported on incorporating additives in PE films such as biopolymers, natural extracts, essential oils, organic acids, enzymes, bacteriocins, fungicides, and others that have the same goal to embed antimicrobial properties of the PE films [3]. The addition of those additives also gives a high contribution to the degradation rate of the film, which lack of studies discussed. Incorporation of zeolites in PE improves the degradation properties of PE because the strong acid sites of zeolites promote the polymer chain cracking (production of low molecular weight hydrocarbon) [4]. Zeolites-silver loaded into PE films function as additives that controlled the release of silver ions from the film to the medium to inhibit the growth of microorganisms, and zeolites-silver did not change the thermal degradation of the film [5]. In other respects, polysaccharide, polypeptides, protein, polynucleotides, and polyester are the natural polymers that had been explored as an agent that able to improve degradation properties but not antimicrobial activities of PE films.

Aloe vera (AV) is a popular material with antimicrobial, antioxidant, and antifungal property, rich in polysaccharides and organic acids [6], [7]. To date, PE and AV have not been formally examined on AV impacts on

the characteristics and mechanical properties of the PE film. A few studies have been conducted on the impact of AV in starch-based film. Researchers used AV as a cross-linker in the starch-based film to improve the properties [7]. AV gel is added into the formula of film for the application of AFP for wrapping or covering easily spoiled food products such as meat or fresh fruits [6]–[8]. AV able to improves the compatibility between starches and plasticiser because the cross-linking occurred between starches molecules due to the presence of organic acid and polyphenolic compounds in AV [8]. Besides, a combination of AV with plasticiser could form a new film that has the potential to become the raw material for a new film [9].

The organic acid in AV increases the hydrogen bonding interaction and enhances the hydrophobic nature of starch. Thus, it led to the plasticising effect that improves the thickness, solubility, molecular weight, thermal properties, and mechanical behaviour of the starch film [10]–[12]. Incorporation of AV with polyvinyl alcohol and citric acid also promotes the tensile strength of the film [7]. AV was even able to increase the thermal degradation of the film [8] at once, affecting the crystallinity of the film [9]. Therefore, AV gel was added into PE film as additives that able to improve the mechanical properties of the film. To the author's knowledge, there is no study conducted on combining PE-based film with AV gel. The PE films were prepared using a melt blending method incorporated with AV gel. The films were then characterised by the presence of the functional group, the appearance or changes of crystalline structure, the degradation temperature profile, thermal analysis, and impact of AV on mechanical properties performance.

METHODOLOGY

Material

Low-density PE (Lotte Chemical, Titans, Malaysia) and extracted AV Gel (Malaysia)

Extraction of AV gel

The AV gel extraction modification method was based on a previous study method [8], [11]. The extraction started with the removal of yellowish liquid from the cut leaves for five to ten minutes. Then, the leaves were cleaned using distilled water, and a paper towel was used to dry it. The leaves were then cut horizontally, and the gel was transferred into a blender. The gel was blended for three minutes and was filtered using cheesecloth to discard the impurities. The permeate was used for blending with PE.

Preparation of the film

Table 1 shows the formulation of the film. The procedure that applied to produce the film was a modified method that consists of three main steps; 1) melt-blended, 2) crushing, and 3) hot press [8], [13]–[15]. Firstly, the PE and AV were pre-mixed in a beaker by manually stirring process for 30 minutes and left for 24 hours in a desiccator. The mixture was then melt blended in a Banbury Thermoo Haake PolyLab Internal Mixer at operating condition of 170 °C, 60 rpm rotation, and ten minutes duration to form a homogeneous solid resin. Secondly, the solid resin was crushed in Rexmac Compact Crusher to break it down into a smaller size that less than 2 mm. Finally, the crushed resin was compressed using Cometech Hot Press at three phases condition; 1) pre-heat phase, the material was pre-heating at 130 °C, 0 psi and ten minutes, 2) hot pressing phase, the material was pressed at 130 °C, 700 psi and ten minutes, 3) cooling phase, next to water tab were flowing through the compression plate at 700 psi until reach room temperature. The film was then peeled off from the steel plate.

Table 1: The Formulation of the Film

Sample name	PE (g)	AV (g)
PE	60	0
PE-AV3	58.2	1.8
PE-AV5	57	3
PE-AV7	55.8	4.2
PE-AV9	54.6	5.4

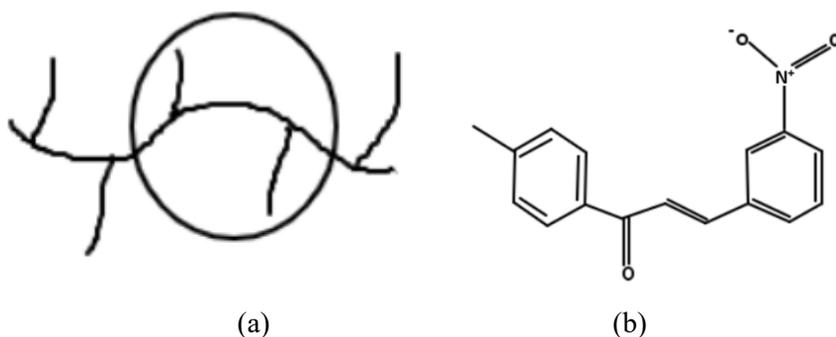


Figure 1: Structure of (a) PE [16] and (b) AV Gel [17]

Characterisation of the film

Infrared (IR) spectra of the films were determined using a Fourier Transformed Infrared Spectroscopy (FTIR) spectrophotometer (Mettler Toledo). IR absorbance spectra were collected at the range of frequency between 640 – 4000 cm^{-1} , running time, 64 scans and with 4 cm^{-1} resolution. Figure 1 shows the chemical structure of PE and AV and will help in determining the presence of new functional groups due to the combination of both materials. The films were characterised using an X-ray Diffractometer (XRD) (PANalytical Aeris). The range of diffraction angles (2θ) was 5-70, and the scanning rate was $2.1786 \times 10^{-4} 2\theta/\text{s}$ for 25 minutes. The XRD equipped with $\text{CuK}(\alpha)$ -radiation filtered by cross-coupled Göbel mirrors at 40kV and 40 mA. The average crystalline size was calculated based on Scherer's Equation and obtained from the diffractograms. The degree of crystallinity also can be calculated using the same diffractogram but based on equation 1.

Differential scanning calorimetry (DSC) was used to analyse the thermal properties of the films. The data taken were the melting temperature (T_m), crystallisation temperature (T_c), enthalpy of fusion/melting (ΔH_m) and enthalpy of crystallisation (ΔH_c). A DSC (Mettler Toledo) was used under the nitrogen atmosphere (50ml/min flowrate). An amount of 5 mg samples was weight, transferred, and sealed in an aluminium pan before undergo three heating processes. The three heating process is also known as cycle process

involved; 1) first heating (25 to 190 °C), 2) cooling (190 to -10 °C) and 3) second heating (-10 to 190 °C) at a heating rate of 20 °C/min. First heating was designed to erase previous thermal history while the cooling imparts a known thermal history. The T_c obtained from the cooling process at the maximum exothermic peak, and the area of the exothermic curve represents the ΔH_c . The second heating was related to real internal differences in the material where T_m obtained from the maximum endothermic peak, and ΔH_m represents by the area of the endothermic curve.

Thermogravimetric analysis was performed using a Thermal Gravimetric Analyser (TGA) Instrument (TGA/SDTA581, Mettler Toledo). Samples approximately 20 mg were subjected under a nitrogen atmosphere (50 ml/min flowrate), at ranging temperature between 25 to 600 °C, and a constant heating rate of 10 °C/min. The weight loss represents the material thermal stability and the fraction of the volatile component. The weight loss was calculated based on equation 2, where: W_i is initial weight, and W_f is the final weight.

Mechanical Properties

A load test was carried out using a Universal Testing Machine (Instron 3382), where the films were strained at a rate of 10 mm/min, room temperature condition. The samples were cut with a length of 100 mm and width 25 mm, following requirements from ASTM D882 and strained at a rate of 10 mm/min, room temperature condition. The U_E was measured from the area under the stress-strain curve until the yield point, while U_T was the area under the curve until the fracture point.

RESULTS AND DISCUSSION

Sample Appearances



Figure 2: Photographs Film of a) PE, b) PE-AV3, 3) PE-AV5, 4) PE-AV7 and 5) PE-AV9

Figure 2 shows photographs of PE-AV film. Clear transparency is demonstrated by PE film. The addition of AV did not have a significant impact on the transparency of the film observed by the naked eyes.

Spectroscopy

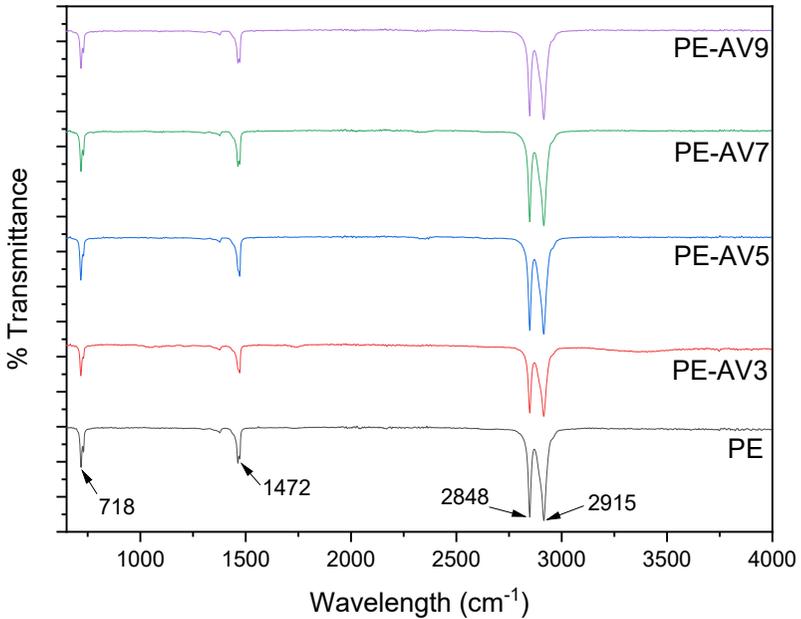


Figure 3: FTIR Spectra of PE and PE-AV Films

Figure 3 displayed the infrared spectrum for PE and PE-AV films at wavenumber between 640 - 4000 cm^{-1} . The same peak shows in all films. Four major peaks occurred at 718 cm^{-1} and 1472 cm^{-1} , which representing strong C-H bending, and medium C-H bending while 2848, and 2915 cm^{-1} (both representing medium C-H stretching). The C-H represents the main chemical structure presence in PE. Amelia *et al.* [18] obtained the same peak representing PE film, where the absorbance bands were located at 2914, 2847, 1470, and 718 cm^{-1} [18]. The typical peaks for AV gel were a broad peak between 3000 and 3600 cm^{-1} representing the O-H stretching and sharp smaller peak at 1635 cm^{-1} representing the bending mode of free water characteristic. Bajer found that the dried AV formed a broader peak of O-H stretching in between 2500 – 3600 cm^{-1} , and a small sharp peak obtained around 2900 cm^{-1} appeared as the medium C-H stretching. Other important peaks also appeared at 1550, 1400, 1200, and 1100 cm^{-1} representing N-O stretching, O-H bending, and C-O stretching, respectively [19]. The fabrication process caused the water to evaporate and caused AV O-H stretching and bending, C-O stretching and N-O stretching disappeared. However, there is a possibility of interaction that happened between these two materials where the sharing peaks obtained at 1472 cm^{-1} (chemical shifting from 1400 peak of AV shifted to 1472 cm^{-1}) and 2915 cm^{-1} (chemical shifting from 2900 peak of AV shifted to 2915 cm^{-1}).

Crystal Structure

Figure 4 represents the diffractogram intensity of PE and PE-AV films obtained from XRD analysis. The amorphous peak of PE appeared at a diffraction angle of 19.6, and two crystalline peaks occurred at 21.42 and 23.66 diffraction angle. Previous research found the same diffraction angle occurred at 20, 21.7, and 24 for pure PE film [20]. The addition of AV into PE films has eliminated all diffraction angles that appeared in PE films, and new diffraction peaks rise at 44.34, 51.76, and 61.73, respectively. These new peaks change the crystalline structure of PE film, and the intensity of diffractogram PE-AV display lower crystalline peak intensity compared to PE film. For PE-AV films, the intensity of the peak decreasing at the AV concentration increased. The presence of the first peak resulted from the amorphous component in the polymer and usually shown by a broad region intensity [20]. The changes in the PE structure proves that AV is the

crystalline structure of PE, and there is chemical bonding between them. From the XRD diffractogram, the crystalline size and degree of crystallinity were calculated and presented in Table 3. From Table 3, the addition of AV had reduced the degree of crystallinity from 84.07% to less than 78%, and the lowest is performed by PE-AV9. AV had disturbed the molecule's range order and reduce the periodicity of PE. The less intensity peak of XRD showed by PE-AV9 had root to an imperfect degree of crystallinity. The average crystalline size obtained presents in Table 3 for each film was between 16.03 – 25.13, calculated based on Scherrer's equation.

Thermal Degradation

Figure 5a displays the TG curves of PE and PE-AV films. It shows that 100% of degradation occurred at a temperature of more than 496 °C for all samples. It can be seen that the introduction of AV reduces the decomposition temperature of the PE. The decomposition temperature (T_d) is almost the same for each sample except PE-AV3. The T_d of PE-AV3 is 238.35 °C, lower value compared to other films which were between 397 and 403 °C. Based on PE-AV3 T_d value, the PE-AV3 film faced the most responsive condition to the heat. However, the mass loss of 50% sample shows by the decomposition temperature at 50% ($T_{d50\%}$) were about almost the same for all samples; PE-AV5 (468.25 °C), PE-AV7 (470.7 °C), PE-AV3 (472.3 °C), PE-AV9 (476.4 °C), and PE (481.9 °C). Even though the thermal stability of PE-AV3 was decreased, the film is still stable at a temperature below 200 °C. This validates that the operating condition during melt blending and hot press technique did not cause any thermal degradation to the films. The presence of AV (additives), making a polymer film thermally less stable [21]. The maximum decomposition rates of samples were analysed by using DTG thermograms, as shown in Figure 5b. It was revealed that only one step involved in the thermal degradation of the films. The DTG thermogram of PE and PE-AV films reveals a peak at maximum temperature (T_{max}) of 470 °C (PE-AV5), 471 °C (PE-AV3) and 478 °C (PE, PV-AV7, and PE-AV9) which represents the decomposition of PE and AV. Similar results obtained when nanoparticles added to PE, the T_{max} did not significantly change [22]. The highest weight reduction was experienced by PE-AV5 (0.61 mg/min) followed by, PE-AV7 (0.6 mg/min), PE-AV3 (0.51 mg/min), PE-AV-9 (0.46 mg/min). Based on the results, it is validated that the modification of PE by the addition of AV does affect the rate of weight reduction.

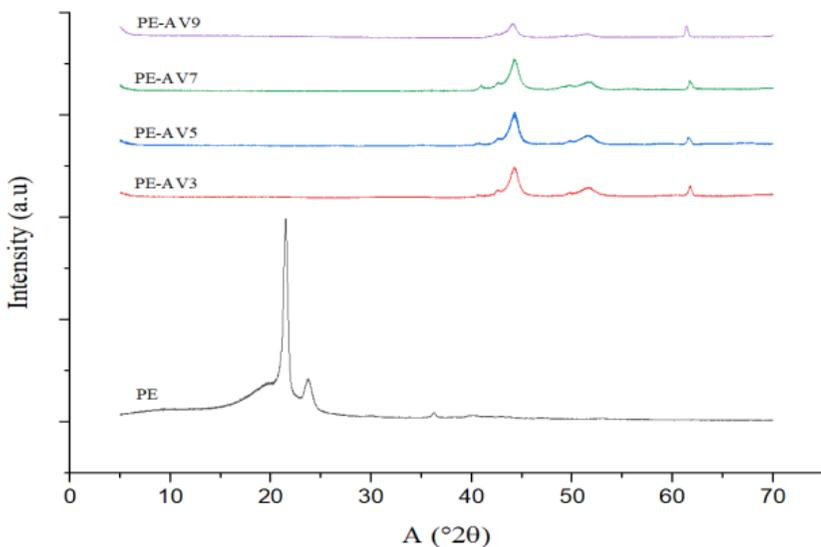


Figure 4: XRD Pattern of PE at Different Concentration of AV gel

Table 3: Crystallise Size and Degree of Crystallinity

Sample	Average Crystallise Size (nm)	Degree of crystallinity (%)
PE	19.32	84.07
PE-AV3	22.01	76.94
PE-AV5	25.13	76.83
PE-AV7	17.80	77.90
PE-AV9	16.03	70.53

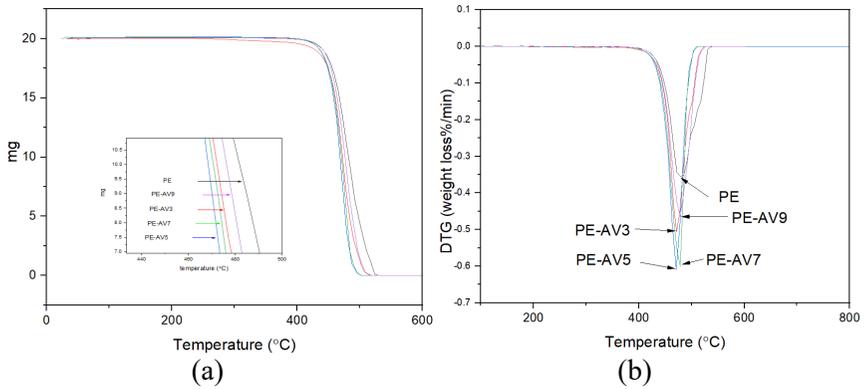


Figure 5: a) Thermogravimetrics (TG) b) Derivative Thermogravimetric (DTG) Curves of PE at Different Concentration of AV Gel

Thermal Analysis

Figure 3a) and 3b) represents the heating and cooling curve for PE and PE-AV films obtained from DSC analysis. The peak derived from the heating curve determines the melting temperature (T_m). In contrast, the peak from the cooling curve gives the crystallisation temperature (T_c), and the value is tabulated in Table 2 along with enthalpy of fusion (ΔH_m) and enthalpy of crystallisation (ΔH_c). The results show that there were no significant changes in T_m and T_c when AV was added into PE film. It justifies that weak interactions present between polymer and AV, as also stated by other researchers [21]. The decrease of T_c might due to low interaction between the hydroxyl group of PE and 5% AV at once increase the flexibility of the polymeric chain. AV did not show huge differences in T_c value, thus indicates the low nucleating effect on PE film. If the nucleating agent had a good result, the crystallinity would increase, and T_m also increased [23]. Linares *et al.* found the same impact of additives to polypropylene film, where weak interaction between molecular segments of the components resulted in high interfacial tension (at the molten stage) lead to no changes in thermal properties of the film (poor dispersion of particles) [24].

The value of ΔH_m was obtained from the area under the heating curve while ΔH_c from the area under the cooling curve. At 5% of AV, there are slightly small changes of temperature for T_c (0.7°C reduction), but somewhat affecting the value of ΔH_c . An abnormal result of ΔH_c obtained at 5% AV, where less energy released to crystallise the material. The thermal properties of PE were influenced by the kinetics of crystallisation during cooling and the reduction of T_c at 5% AV. When the T_c decreased, the number of nuclei per unit volume also decreased [23]. The slight decrement of T_c occurred at 5% AV, and simultaneously, the ΔH_c reduced. This phenomenon suggests that at 5% AV, there is a possibility of self-nucleation of PE being destroyed and saturated. There is a possibility of the macromolecular movement speeding up and prevent the growth of polymeric crystals around the small nuclei [24].

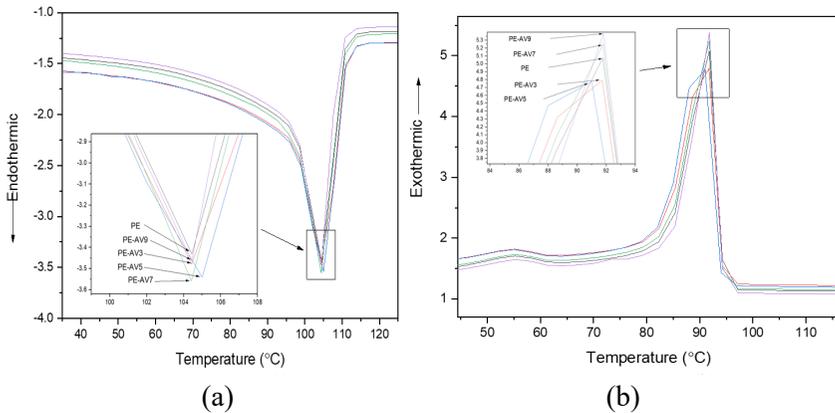


Figure 6: DSC Curve of a) Heating and b) Cooling for PE and PE-AV Films

Table 2: DSC Data of PE-AV Films

Sample Film	T _c (°C)	T _m (°C)	ΔH _m (J/g)	ΔH _c (J/g)
PE	91.76	104.44	133.57	88.56
PE-AV3	91.72	104.43	139.96	85.01
PE-AV5	91.00	105.00	123.18	57.16
PE-AV7	91.78	104.42	141.92	92.43
PE-AV9	91.80	104.44	134.63	84.62

Mechanical Properties

Stress-strain curve for the films is shown in Figure 7. The ductile stress-strain profile is displayed by each film, where the stress increase until it achieved a point that the stress stable for a certain time and it sustain the elongation more than 50% before slowly ruptured. The maximum breaking extension is achieved by PE-AV7, followed by PE-AV5, PE, PE-AV3, and finally PE-AV9. Table 4 summarises the TS, EAB, YM, UE, and UT. PE-AV3 is having the highest value of TS and YM, while PE-AV7 has the highest elongation at break (EAB), 77.67%. Increasing TS and YM of PE-AV3 could be due to low free volume and the presence of cross-linking with PE molecular structure. The possibility of the reaction occurred between AV and PE, also supported by the crystalline structure changes shown by XRD results.

However, the reduction of TS occurred with more amount of AV might be due to AV facilitated the sliding of PE chains and lowered the secondary linking forces between the polymer chain. The finding by Jusoh [24] stated that the addition of other materials that able to cause a lubricating effect would reduce the TS of PE film. These results were supported by other research that found the presence of other material in PE disturbs the PE chain structure at once reduced the mechanical properties [21]. The EAB value of PE-AV film increased compared to PE film but sudden drops at 9% AV. This might occur due to the high amount of AV cause poor adhesion between AV and PE. This result supported by the XRD peak for PE-AV9 that shows low-intensity peak if compared to other PE-AV films. The interfacial adhesion between two polymer matrix plays an important role in the EAB of the polymer [25]. Usually, high TS polymer will give high stiffness and toughness to the material because of the intermolecular forces high. The

stiffness could be related to the EAB value. Increasing EAB shows low stiffness of material where the presence of 7% AV reduces intermolecular forces within the polymer chain, hence improve the mobility and flexibility of the polymer structure [21]. The highest value of U_E and U_T was obtained by PE-AV3, and the presence of AV shows better value compared to PE alone except for PE-AV9. Similar results proved that the addition of copolymer or other filler at a certain percentage between 1.5 – 5% could improve the toughness characteristic of PE film [26]. The U_E and U_T values were affected by phase morphology, chain structure, and interfacial adhesion [27].

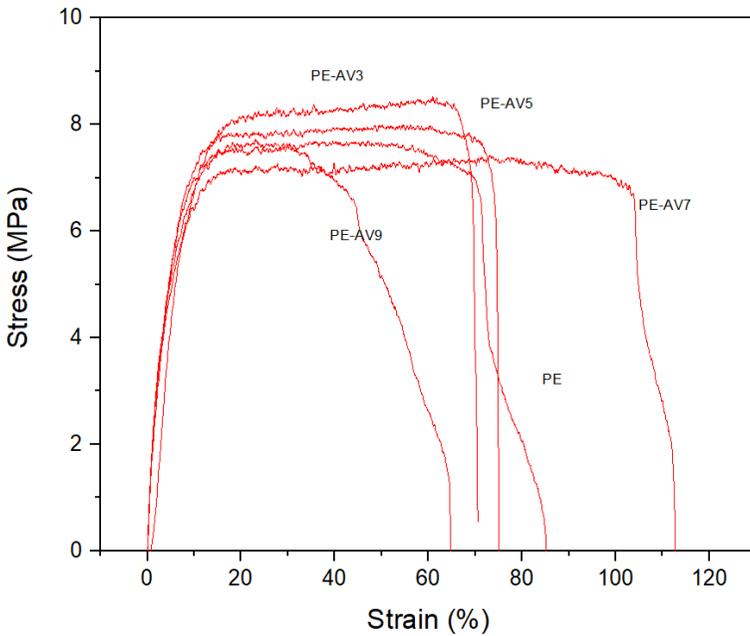


Table 4: Mechanical Properties of the Films

Film	TS (MPa), σ	EAB (%)	YM (MPa), ϵ	Modulus of Resilience (MJ/m ³), U_E	Modulus of Toughness (MJ/m ³), U_T
PE	7.79	52.20	122.03	9.87	279.76
PE-AV3	9.09	55.60	124.48	17.70	492.73
PE-AV5	8.31	54.67	95.58	11.36	361.46
PE-AV7	7.80	77.67	103.41	16.29	428.47
PE-AV9	8.00	18.20	121.30	7.83	126.54

CONCLUSION

The PE films consist of AV as an additive were successfully prepared using melt blending and hot press technique. PE-AV3 was the best film with the highest tensile strength. Crystal structure and mechanical properties show significant differences after the addition of AV to PE films. The specific concentration of AV gave the highest value of the TS, YM, UE, and UT and contributing to lowering the thermal degradation temperature. There were no huge differences in the functional group peaks (spectroscopy) and thermal properties (T_m, T_c, and enthalpy). The primary outcome shows that TS plays an essential role in this study because it indicates that low PE content with a certain amount of AV can improve the performance of TS. Therefore, AV has excellent potential to be one of the additives in the future development of AFP.

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