

Significant impact of melt-blend and hot-press technique operating condition on polyethylene/thermoplastic starch/aloe vera gel film properties

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Abstract

Melt-blend and hot-press techniques were among the most popular methods for producing polyethylene/thermoplastic starch (PE/TPS) polymer blends. However, different operating conditions contributed to the various quality of film made. Thus, this paper focuses on determining the most suitable operating conditions involving melt-blend and hot-press techniques on specific PE/TPS/aloe vera gel (AV) film formulations. Thus, this study determines the best operating condition for producing PE/TPS/AV blend. One-factor-at-a-time (OFAT) method was implemented under six operating conditions: different melt-blending temperatures, pre-heating, hot-pressing pressure, temperature and duration, and cooling application. The result demonstrated that the best melt-blending temperature was 170 °C, having the highest tensile strength (TS) performance. Pre-heating application reduced the thickness, which is suitable for applying single-used plastic and more economical than thicker film. Importantly, this study found the lowest pressure to obtain a smooth surface of film without stickiness during the peeling process from the steel plate after applying the hot-press technique. The same condition faced at 130 °C, hot-pressing temperature produced a smooth and clear film structure. The pro-longed hot-pressing duration reduced the bonding strength of the polymer matrix. Finally, a fast-cooling rate enhances the TS of the film. Therefore, the melt-blending and hot-pressing operating conditions significantly impact the physical and mechanical properties of PE/TPS/AV film. The results of this study are valuable for understanding that each formulation needs a specific processing condition essential as an initial assumption in simulation processes.

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1.0 Introduction

Polyethylene (PE) is the primary raw material used globally in producing single-used plastic because highly resistant to the chemical and harsh environment (Datta & Halder, 2018; Min Min et al., 2008; Sabetzadeh et al., 2017). However, due to its long polymeric structure, the ability of PE to degrade upon being buried in soil, water, or an open environment was inferior (Al-Salem et al., 2019). Thus, it threatens the world's environmental condition due to single-used plastic accumulation. Therefore, biodegradable-based plastic was introduced, made from starch. Starch had been considered a biodegradable material consisting of amylose and amylopectin, making it suitable for replacement for PE (synthetic polymer). The starch-based biopolymer can transform into thermoplastic starch (TPS) upon adding a plasticiser and operated at

high temperatures to enhance the starch's capability as a polymer material (Ibrahim et al., 2020). However, TPS alone as plastic packaging material did not support industrial needs because the film's poor mechanical and barrier properties have been diminished (Gutiérrez & Álvarez, 2016; Martins et al., 2018).

Blending PE and TPS was started 30 years ago to improve PE's degradation ability and increase TPS's mechanical properties (Datta & Halder, 2018). The PE/TPS polymer blend is commonly produced for food packaging applications as single-use plastic (Datta & Halder, 2018, 2019; Mehmood et al., 2016). The film can be blended using different methods, such as using a polymer reactor (Raman & Jinwoong, 2011), polymerization method (Tamada et al., 2004), and melt-mixing method (Amigo et al., 2019). The melt-mixed process emphasised a batch mixer equipped with a Banbury mixer. The Banbury mixer produced

the solid resin, which was transported to a different machinery piece to form a film sheet. The melt-blending process's advantages were toxic-free, controllable residence time, controllable temperature, and controllable mixing conditions (distributive and dispersive). However, the adhesive material sticks to rotors and mixing chamber walls. The resin from the melt-mixer was transferred to compression molding (hot-press) to convert it into a thin film.

Many studies have used melt-blending and hot-press techniques to blend PE and TPS to produce a thin film (Jantanasakulwong et al., 2018; Nguyen et al., 2016a; Sarifuddin et al., 2014). Nguyen et al. (2016a) set the melt-blending mixer for blending low-density PE with cassava starch at 7 minutes, 60 rpm rotational speed, and 160 °C operating temperature, while the hot-pressing condition was 160 °C, 1500 psi with a cooling process applied. Jantanasakulwong et al. (2018) blended thermoplastic corn starch and polyethylene-grafted-maleic anhydride at 160 °C for 5 minutes with the hot-pressing condition of 160 °C for 3 minutes. Limited papers combined both techniques in producing a thin film based on PE/TPS blend. Most reports did not focus on the effects of processing conditions on the performance of the PE/TPS blend with the addition of a compatibiliser. The TPS preparation is essential since the gelatinization process occurs with heat at a specific temperature because the high temperature might disrupt the starch molecules. Thus, temperature plays a critical role in sustaining starch components' characteristics.

Furthermore, the addition of additives such as polyethylene grafted maleic anhydride, cellulose nanofibers, citric acid, and AV gel has been studied to improve the strength ability of PE/TPS film (Ahmadi et al., 2018; Martins et al., 2018; Nguyen et al., 2016b; Abd. Karim et al., 2021). AV gel has previously been used in film packaging development based on biopolymer materials such as starch and gelatin (Chin et al., 2017; Mallick et al., 2020). The amino acids and polysaccharides in AV gel helps improve the polymeric structure of starch-based polymeric structure (Kanatt & Makwana, 2020). Unfortunately, limited study have reported the operating conditions of producing film sheets with AV gel.

Therefore, it is necessary to determine the best operating conditions to keep the film's full benefit. The change in melt-blending and hot-pressing parameters and its effects on the mechanical properties of PE/TPS/AV blend film has not yet been explored.

Thus, this study investigated the impact of different operating conditions during melt-blending and hot-press techniques on the film's thickness, physical appearance, and mechanical properties.

2.0 Methodology

2.1 Material

Low-density polyethylene (Density: 0.915 g/cm³) obtained from Lotte Chemical, Titans, Malaysia. The thermoplastic starch was prepared based on the combination of glycerol (Molecular weight: 92.09g/mol; Chemiz (M) Sdn. Bhd.) and soluble potato starch (Bendosen, Malaysia) obtained from Laupik Chemical. Chemieconnex, Malaysia, supplies the Aloe Vera gel.

2.2 Methods

Formulation used for PE/TPS/AV film blend was 18.91 g of PE, 16.54 g of TPS and 0.84 g of AV gel which was the optimum formulation obtained from the previous work by Abd. Karim et al. (2021). The film preparation procedure started with pre-mixed 70% starch with 30% glycerol and kept overnight. Then, the starch/glycerol mixture and AV gel were added into the Thermo Haake PolyLab Internal mixer to form a resin at different melt-blending mixing temperatures to blend all materials to produce a solid resin. Mixing speed of 60 rpm, and mixing duration of 20 minutes, were kept constant throughout the melt-blending process. The resulting resin is then transferred to Rexmac compact crusher to form smaller resin to improve the heat transfer process to the resin during hot-pressing. The crushed resin (smaller size) was then compressed using Cometech hot-press machine. Table 1 shows the effects of the operating conditions monitored for this work.

Table 1: The parameter range applied to the different operating conditions

Operating condition	Parameter range	Properties monitored
Melt-blending mixing temperature	120–190 °C	TS, EAB, Th
Pre-heating technique	with and without pre-heating	Th
Hot-pressing pressure	700, 800, 900, 1000, 2000 psi	Phy App, Th
Hot-pressing temperature	130, 150, 170 °C	Phy App
Hot-pressing duration	5, 10, 15, 20 minutes	TS, EAB, Th
Cooling technique	With and without cooling	TS, EAB, Th

TS, tensile strength; EAB, elongation at break; Th, thickness; Phy App, physical appearance

Tensile strength (TS) and elongation at break (EAB) of the film were measured using Universal Testing Machine (Instron 3382) at a 25 mm/min strained rate at room temperature. The dimension of the film was 100 mm in length \times 25mm in width, based on ASTM D882. The thickness was measured using an IP 65, 700-118-30, Mitutoyo digital thickness gauge (0–1 in., 0.0001 mm), with the readings closest to around 0.0001 mm (1 μ m) at eight random sites. The physical appearance was observed using the naked eye and captured using a commercial Redmi Note 9 Pro, commercial hand phone camera, Xiaomi, following the same approach used by a prior study to determine the film's transparency (Panrong et al., 2020).

3.0 Results and discussion

3.1 Effect of melt-blending temperature

3.1.1 Torque

The torque measurement evaluated the PE/TPS/AV processability by representing the melt hindrance during the blending operation. Fig. 1 shows the torque obtained from the mixing process for combining PE, TPS, and AV using an internal Banbury mixer at a different temperature range between 120 to 190 °C for 20 minutes at 60 rpm. For mixing temperatures of 120 to 150 °C, higher torque variations occurred to achieve stability compared to 160–190 °C, which has fewer variations over more time exposure to the mixture. Fewer variations were observed at higher temperatures due to all materials achieving their melting condition and blending appropriately. Plus, all curves show duration reduction at a higher temperature for the film to be homogeneous fast. A similar observation was explained by Patti et al. (2020), which observed the non-monotonous torque, allowing the polymer's melting to be blended with the fibre for less than 10 minutes before achieving torque stability at 170 °C. The torque value needs to be stable for a particular time before the mixing process ends to ensure the mixing process achieves the best homogeneity condition. All film has loading torque from 0.0 to 4.5 Nm and maximum torque between 3.7 to 4.5 Nm (almost consistent).

Table 2 lists extracted data from Fig. 1, showing the samples' stability time, stability torque, and maximum torque. From Fig. 1, during the initiation of the mixing process, torque rapidly increased and achieved maximum due to the uniformness of the particles corresponding to the polymer melting. After a

Table 2. Maximum torque, stable torque, and stability time for mixing PE/TPS/AV film.

Melt-mixer temperature (°C)	Starting stable time (min)	Stable torque (Nm)	Maximum torque (Nm)
120	20.0	1.7	4.5
130	17.8	0.6	4.0
140	16.2	0.4	3.9
150	15.6	2.7	4.3
160	13.5	3.1	4.1
170	12.4	3.2	4.2
180	12.2	2.8	3.9
190	11.7	2.4	3.7

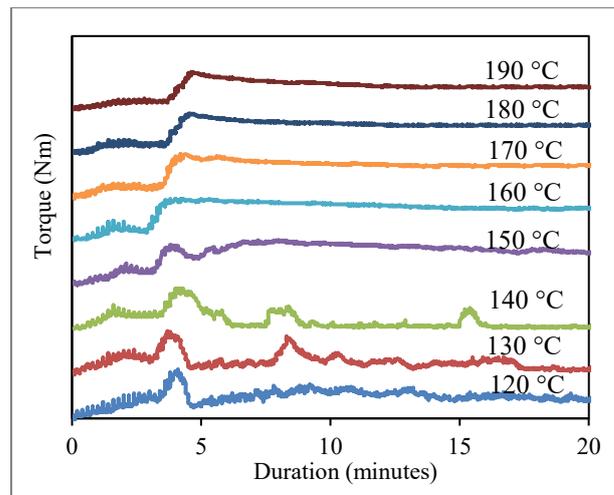


Fig. 1: Torque obtained for the mixing process

homogenous blend, torque variations became more stable; with the temperature of 120, 130, and 140 °C, the torque values became more stable of 1.7, 0.6, and 0.4 Nm, respectively, whereas at 150 to 170 °C, greater torques values were observed. The higher variation of torque value could also be observed in Fig. 1 at the lower range of temperature, showing poor stability of homogeneity in the mixture. The maximum torque is shown at 120 °C and decreases over increasing temperature. The maximum torque was 4.5 Nm and fell over the increased temperature applied. The results agree with Saddem et al., (2019), who stated that higher instability was linked to the amount of fibre incorporated into the polymer matrix at lower temperatures. This lead to difficulty in releasing generated heat inside the compound (Tomaszewska et al., 2011).

3.1.2 Thickness, tensile strength, and elongation at break

Fig. 2 represents the effect of mixing temperature on the thickness, TS, and EAB of PE/TPS/AV film. The thickness of each film was 0.20 ± 0.011 mm,

almost consistent, proving that temperature did not significantly affect the thickness of the film. The TS increased as increasing temperature from 120 to 170 °C. Unfortunately, the TS and EAB decreased when mixing at more than 170 °C temperatures. The increase in temperature enhanced the fluidity of the material's melting behaviour, which facilitated the inclusion of each constituent to create a polymer blend matrix. TS increased, especially up to 170 °C, because of more contact surface area. The same prediction was observed when polypropylene/woven composite tensile enhanced with higher operating temperature due to better matrix fluidity that aids matrix impregnation that increased contact surface area (Rokbi et al., 2020). The reduction of mechanical properties over 170 °C might be due to changes in starch structure, making the matrix less dense. This agrees with a previous study that found the TS of the thermoplastic starch-based film decreased at higher temperatures (Zakaria et al., 2018). Therefore, a mixing temperature of 170 °C was chosen for the following process because it has the highest TS and EAB.

3.2 Effect of the pre-heating process on the film thickness

Pre-heating was applied to the sample to enhance early heat distribution for 10 minutes (Ahmadi et al., 2018). The PE/TPS/AV thickness with pre-heating was 0.2070 ± 0.0126 mm and 0.4484 ± 0.0185 mm for samples without pre-heating, an increment of 100% compared to with pre-heating applied. The observed reduction in thickness indicates that viscoelastic materials experienced a decrease in viscosity, resulting in improved flow characteristics and reduced thickness. The earlier work demonstrated that pre-heating the composite resin reduced viscosity and enhanced fluidity of visco-elastic materials, thus decreasing thickness by 30% (Bhopatkar et al., 2022). A similar argument has been given: preheating increases the composite's ability to flow, thus decreasing its thickness (Dionysopoulos et al., 2014).

3.3 Effect of hot-press pressure on the physical appearance and thickness of the film

Based on the literature review for the hot-pressing procedure for TPS/PE blend, the range applied for hot-press pressure was between 508 to 3500 psi depending on the metal sheet used to convert the resin into a film sheet (Bagheri et al., 1997; Datta & Halder, 2019; Nguyen et al., 2016a; Raghavan & Emekalam, 2001). Table 3 shows the physical appearance and thickness

Table 3: The physical appearance and thickness of PE/TPS/AV film at different hot-press pressure

Pressure (psi)	Physical appearance	Comments	Thickness (mm)
2000		Ruptured film in the middle	0.09
1000		Ruptured film in the middle	0.11
900		Ruptured film in the middle	0.11
800		Ruptured film in the middle	0.11
700		Easily peeled off from the plate	0.18

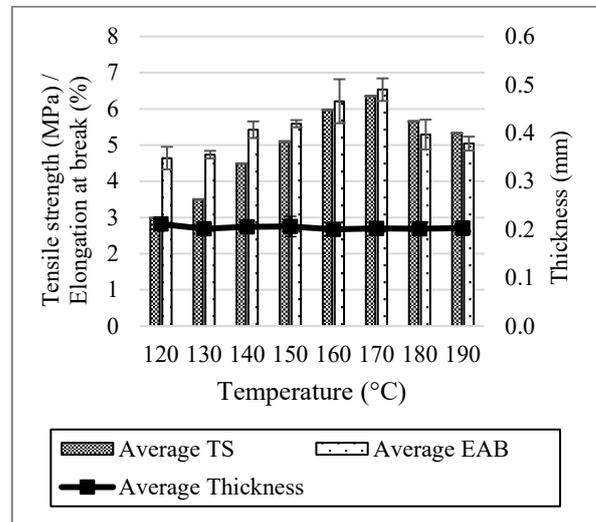


Fig 2: The film's thickness, tensile strength (TS), and elongation at break (EAB) at different temperatures

of the film after pressing at different pressure (between 700 to 2000 psi) with a mixing temperature of 170 °C and a hot-press temperature was 130 °C. The thickness sample for 700 psi was 0.18 mm, 63.64% higher than the thickness for the pressure applied more than 800 psi, which was between 0.09 and 0.11 mm. A similar result was obtained by Wang et al. (2022), in which the thickness of the titanium mesh-based membrane

electron assembly gradually becomes thinner upon pressure increment. Zou et al. (2021) also found that the increment in pressure reduces the thickness of the membrane. According to Tharazi et al. (2017), higher processing pressure damages the fiber and leads to matrix starvation. The excessive force applied to the film quickly leads to over-compression, which ruptures in the middle at higher pressure (>800 psi) because of the film's low thickness. At 700 psi, the film can easily be peeled off from the plate, and an excellent thin film with a thickness of 0.18 mm was obtained.

3.4 Effect of hot press temperature on the physical appearance of the film

Most of the previous studies used mixing temperature as the hot-press temperature. Table 4 shows the sample's appearance at different temperatures applied on hot-press machines. This study shows ruptured film obtained when 170 and 150 °C pressing temperatures were applied at 700 psi. A good film sheet was produced when the hot-press temperature was at 130 °C because the film could be peeled off easily from the plate. The increasing temperature caused the film to become softer, reducing the internal resistance and rupturing. When the hot-pressing time was fixed, the bonding strength initially increased and then decreased as the hot-pressing temperature increased. This may have resulted from the acceleration of molecular thermal movement and water evaporation during hot-pressing, promoting hydrogen bonding forces' formation. A similar result was observed in a previous study (Zou et al., 2021). Therefore, it is imperative to select the appropriate temperature during hot pressing.

3.5 Effect of hot press duration on thickness, tensile strength, and elongation at break

Fig. 3 shows the effect of hot press duration on the thickness, TS, and EAB of PE/TPS/AV film. The different hot-press duration was applied to the film with a mixing temperature of 170 °C, pressing temperature of 130 °C, hot-press pressure of 700 psi, and pre-heating process. Based on the literature, 3–20 minutes of hot-pressed duration had been applied in the previous study. Thus, this paper used 5, 10, 15, and 20 minutes as pressing periods. The thickness of the film decreased upon higher duration applied. As the time for hot-pressing increased, sufficient curing at the surface and core of the polymer matrix was obtained. Thus, the polymer matrix's flowability to disperse and

Table 4. The physical appearance of the film at a different hot-press temperature

Hot-press temperature (°C)	Physical appearance	Comments
170		Ruptured film in the middle
150		Ruptured film in the middle
130		Easily peeled off from the plate

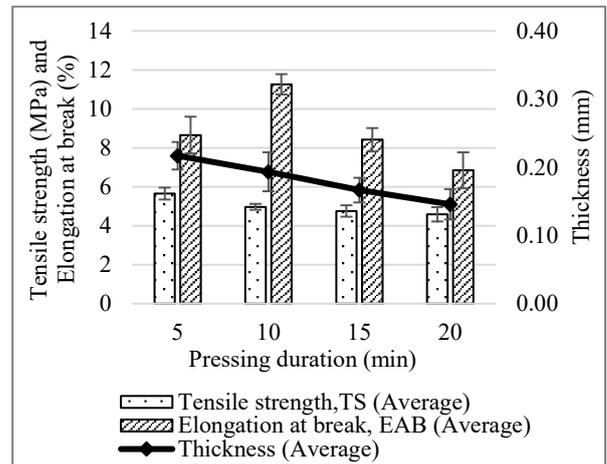


Fig. 3: The thickness, tensile strength (TS), and elongation at break (EAB) of PE/TPS/AV film at different pressing durations

form a thinner film improved. The long hot-pressing time it led to brittle plywood, which reduced the bonding strength of the polymer matrix due to the curing of the polymer matrix being incomplete. This agrees with Wei et al. (2021), which found that the bonding strength reduced after adding more hot pressing time. The TS of the film also decreased upon an increment of hot-pressing time, which is simultaneously affected by the film's thickness. Transferring heat from the surface to the polymer matrix core takes a specific time, but prolonged hot-pressing time reduces bonding strength. Tharazi et al. (2017) also found TS of long kenaf fiber blended with polylactic acid reduced upon an increment of hot-pressing duration. Regarding EAB, the values increased in the hot-press duration and decreased as more time was applied. This means that prolonging hot

pressing time would not promote the free space volume properties of the polymer matrix.

3.6 Effect of cooling technique on TS, EAB, and thickness

As shown in Fig. 4, the cooling technique did not impact the thickness of the film. However, the tensile became lower when no cooling was applied, from 4.97 to 4.11 MPa. The EAB increased from 11.26% to 14.7%. Higher TS is obtained because a higher cooling rate decreases the molecules' size, while slow cooling transforms into soft, coarse, and less dislocated phases. Decreased film density caused by fast cooling rates reduced film stiffness, lower TS, and higher elongation at break. Herlambang & Anando, (2020) stated any processing condition that causes faster cooling will improve the impact strength.

3.6.1 Summary of results

Table 5 sums up the findings of this study. Using a melt-mixing temperature of 170 °C, this temperature is ideal for producing the strongest PE/TPS/AV film. Pre-heating was continued since the thickness remained stable from 0.2023 to 0.2070 mm, and the removal of the pre-heating process raised film thickness by 100%. Even if the thin film is better with a smaller thickness, the TS must still be considered. Pressure and temperature were maintained at 700 psi and 130 °C for the hot-pressing process, which was done to preserve a smooth surface without tearing after peeling off the steel plate. The cooling process was chosen as the process improved the TS of the film, and thickness experienced no significant changes.

4.0 Conclusion

Based on the operating circumstances investigated in this work, the operating condition for PE/TPS/AV has been established. The chosen operating condition for the mixing process of PE/TPS/AV was 170 °C, 60 rpm, and 20 minutes. The pre-heating process was applied for early heat distribution for the hot-pressing condition, which helps the material receive consistent heat before pressing is applied. The temperature for the hot press was 130 °C, 10 minutes of pressing time, with 700 psi pressure applied. The film then was confirmed to undergo tap water cooling technique as cooling affects the film's mechanical properties. The film produced at the decided parameters has 4.97 MPa TS, 11.2% EAB and 0.19 mm thickness. Thus, it can be an alternative to replace 100% PE-based plastic.

Table 5: Summary of results

Operating conditions	Best operating parameter	Results
Melt-blending temperature	170 °C	0.2023 ± 0.0116 mm, 6.35 ± 0.686 MPa, 6.53 ± 0.3123 %
Pre-heating process	With pre-heating,	0.2070 ± 0.0126 mm thickness
Hot-pressing pressure	700 psi	0.18 mm, and a very smooth surface obtained
Hot-pressing temperature	130 °C	Smooth surface obtained
Hot-pressing duration	10 minutes	0.1934 ± 0.0286 mm, 4.97 ± 0.1535 MPa, 11.2 ± 0.5225 %
Cooling technique	Using tap water,	0.1934 ± 0.0286 mm, 4.97 ± 0.1535 MPa, 11.2 ± 0.5225 %

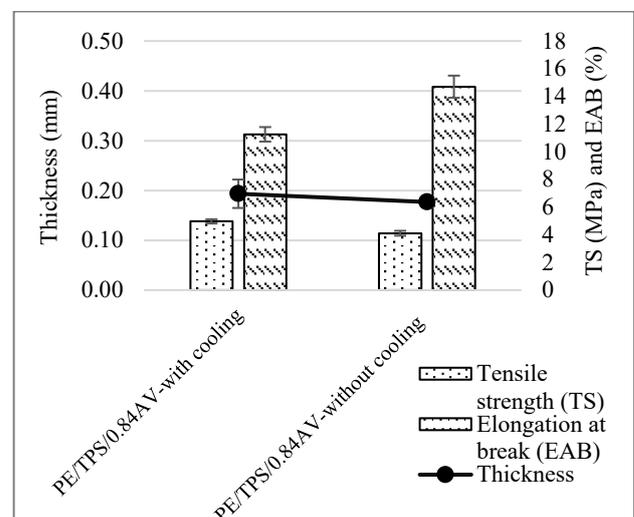


Fig 4: The thickness, tensile strength (TS), and elongation at break (EAB) of PE/TPS/AV film break at different cooling techniques

Contribution statement

Siti Fatma Abd Karim: Conceptualisation, methodology, investigation, formal analysis, data curation, writing – original draft; **Junaidah Jai:** Validation, writing-review & editing, visualisation, supervision; **Rabiatuul Adawiyah Abdol Aziz:** Project administration and resources. **Ku Halim Ku Hamid:** Validation, writing-review & editing, visualisation, and supervision. All authors have read and agreed to the published version of the manuscript.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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