Thermal and mechanical properties of polyethylene-starch based film incorporated with crude palm oil

S F A Karim¹, J Jai¹, K H K Hamid¹, and F N Norhisam¹
¹Fakulti Kejuruteraan Kimia, UiTM Shah Alam, 40450 Selangor, Malaysia

sitifatma9633@uitm.edu.my

Abstract. The present study reports the changes in thermal degradation, thermal properties, and mechanical performance of polyethylene (PE) - thermoplastic starch (TPS) based film incorporated with crude palm oil (CPO) as a plasticizer. The films were prepared using the melt blending and hot-press technique. Then, it was characterized using a Thermogravimetric analyzer, Differential Scanning Calorimetry, and Universal Testing machine to determine the film's thermal degradation, thermal properties, and mechanical properties. TPS in PE film had reduced the thermal stability; however, CPO gives small changes to it. The amorphous phase of TPS reduced PE film's crystallinity. Besides, CPO provided a lubricating effect and improved the crystallinity of PE/TPS film. Unfortunately, the tensile strength and elongation at break keep decreased with an increase of CPO content. The thermal decomposition and kinetic analysis using a thermogravimetric analyzer on one film (PE/TPS/CPO) at three different heating rates (7.5, 10.0, and 12.5 °C/min) were investigated. Friedman isoconversional model-free method was used to calculate the activation energy of the film. The thermal decomposition of materials in the film increased with the increasing heating rate, and the average computed activation energy obtained was 364.68 kJ/mol.

1. Introduction
The combination of polyethylene (PE) and starch to produce a thin film had been studied by many researchers to support each other weaknesses; PE slow degradation has been improved by starch, while starch's low mechanical properties enhanced by PE [1–3]. Both materials have their advantages. Starch is very cost-competitive, abundant supplies, biodegradable, and has a film-forming character [4–7]. The reduction in mechanical properties of a starch film because of high water sensitivity (very hydrophilic) at once also reduced the barrier of the film [8,9]. Therefore, starch needs another polymer to enriched the hydrophobic character. PE is a synthetic polymer used as raw material, especially in packaging applications. A common type of PE used for developing a thin film was linear density with a good character that is very lightweight, low cost, excellent processability, and high resistance to the harsh environment [10,11]. The high resistance characters towards microbial attack for degradation of PE caused a low degradation rate of PE at once plastic accumulation waste increased [12].

Starch without plasticizer cause limitation in undergoing process at high temperature. Plasticizer transformed starch into thermoplastic properties to withstand the processing condition and improved the film's flexibility and plasticity [13]. PE minimum melting temperature normally around 115 °C. The commercial plasticizer used was phthalate, not suitable for food film packaging because it is harmful to human health, while non-phthalate plasticizers caused leaching when compounded with polymer [13]. Glycerol was a common plasticizer applied in starch that reduced the intermolecular forces and changed its thermal properties [14]. Many researchers studied the addition of glycerol into PE-starch-based film,
but unsatisfying results were obtained [4,15,16]. Crude palm oil (CPO) was then introduced as a bio-plasticizer since treated, and modified palm oil was a high cost [13]. CPO able to provide a lubricating effect and form a homogeneous and uniform distribution in polypropylene at room temperature [17].

The polymer material is degraded due to many factors such as humidity, UV light, heat, and ozone, and those conditions can be represented by the heat processing process that is commonly applied by the thermogravimetric method. Thermogravimetric analysis was the most thermal degradation studies to estimate thermal stability and predict the polymeric material's possible lifetime [18]. Kinetics parameters calculated from the thermogravimetric analysis are essential in providing information to design the pyrolysis reactor. The kinetics parameters help in understanding the reaction behaviour and help in developing the thermochemical process. The non-isothermal method is commonly applied because the model fit data is more reliable and straightforward [19]. Isoconversional model-free model like Friedman's method was a type of model-fitting method used to obtain activation energy at the best statistical fit condition.

A few studies were conducted on thin film's thermal stability and mechanical performance to prove that CPO's function is comparable with a synthetic plasticizer [20,21]. Therefore, this paper studied the impact of CPO on thermal degradation, thermal properties, and the film's mechanical properties. The film then consists of PE/TPS/CPO being proceeded on thermal degradation and kinetic analysis using the Friedman method. Plus, there is a limited report using the Friedman method on PE/TPS film on determining the pyrolysis reaction kinetics.

2. Methodology

2.1. Materials
Low-density polyethylene (LDPE) as the synthetic polymer (Lotte Chemical, Titans), crude palm oil as additional plasticizer (Free Fatty Acid: 4.00%, MPOB), potato soluble starch as biopolymer (Bendosen), and, glycerol as primary plasticizer (MW: 92.09, Chemiz).

2.2. Film’s preparation

| Sample Name | Composition |   |   |
|-------------|-------------|---|---|
|             | LDPE (g)    | TPS (g) | CPO (g) |
| Film-1      | 30.00       | 6.00    | 1.00    |
| Film-2      | 30.00       | 15.00   | 1.00    |
| Film-3      | 30.00       | 6.00    | 4.50    |
| Film-4      | 30.00       | 15.00   | 4.50    |
| Film-5      | 30.00       | 10.50   | 2.70    |

The mixing of the raw material and thin film formation followed the previous study [22]. The process started with drying starch and PE in the oven for 24 hours. Glycerol was then mixed with starch to form a thermoplastic starch (TPS) at 70:30 ratios. The blending process of PE, TPS, and CPO was melt-blended in a Thermo Haaker Polylab internal mixer. The operating condition was 160 °C, 60 rpm speed, and duration between 5 to 15 minutes, depending on the torque stability. Each ingredient was added gradually into the mixer with the rotor speed that also rises slowly (5/15/30/45/60 rpm) to improve the mixing process. Before being compressed, the blends were crushed using Rexmac Compact Crusher to obtain a size of approximately less than two mm to improve the heat transfer surface area during the next step. The compression moulding method used to convert crushed blend into a film by Cometch Hot Press at 130 °C, 700 psi for 10 minutes before being cooled by cooling water to room temperature. Lastly, the film peeled off from the steel plate by using forceps. Table 1 represents the formulation of the prepared films.
2.3. Thermal degradation, thermal and mechanical properties

The films' thermal degradation was investigated using Thermal Gravimetric Analysis (TGA) instrument (TGA/SDTAS581, Mettler Toledo) 50 mL/min gas flow rate and 10 °C/min heating rate. The nitrogen atmosphere was chosen at a temperature between 25 °C to 600 °C. The different heating rates applied only to Film-5 at 7.5, 10, and 12.5 °C/min to investigate thermal decomposition at different heating rates and determine the kinetics parameter. Mettler Toledo Differential Scanning Calorimetry (DSC) was used to perform thermal properties analyses with the operating conditions of 50 mL/min gas flow rate and 20 °C/min heating rate under nitrogen atmosphere. Cycle heating process applied started with the first heating was performed at 25 °C to 190 °C, followed by cooling to -10 °C and second heating to 190 °C. DSC conducted to determine the melting temperature (T_m), crystallization temperature (T_c), enthalpy of fusion (ΔH_m), enthalpy of crystallization (ΔH_c) and degree of crystallinity (X_c). The X_c was calculated using equation (1):

\[ \% \ X_c = \frac{\Delta H_m}{w \times \Delta H_{m0}} \times 100 \]  

where ΔH_m is melting enthalpy obtained from DSC thermograms, ΔH_{m0} is the melting enthalpy of 100% crystalline LDPE (293 J/g), and w is the weight fraction of LDPE in the sample. Finally, the tensile strength (TS) and elongation at break (EAB) were measured using a Universal Testing Machine. A load of 2.5 kN and a crosshead speed of 25 mm/min were applied, according to ASTM D882.

2.4. Thermal and kinetics analysis

The different heating rate applied to Film-5. The raw data obtained from TGA analysis was the weight loss (w) concerning temperature (T) and weight loss rate (dw/dt) concerning T. Based on the data, the decomposition temperature and activation energy (Ea) can be determined. For E_a's determination, a kinetics model equations based on isoconversional principle used named as Friedman method. The films' reaction rate during non-isothermal degradation is obtained by the differential equation, as in equation (2). The α(t) can be obtained from equation (3), representing the ratio of the sample's mass loss at time t and total mass during the process. A linear heating rate (β = dT/dt) used for a non-isothermal condition had converted equation (2) to equation (4). Equation (5) was representing equation (4) after integration and approximation.

\[ \frac{d\alpha}{dt} = A \exp\left(\frac{E_a}{RT}\right) \cdot f(\alpha) \]  

\[ \alpha(t) = \frac{w_i-w_f}{w_i-w_f} \]  

A = degree of conversion, w_i = initial weight, w_f = weight at time t, w_f = final weight

\[ \frac{da}{dt} = \frac{A}{\beta} \exp\left(\frac{-E_a}{RT}\right) \cdot f(\alpha) \]  

\[ g(\alpha) = \int_0^T \frac{da}{dt} = \frac{A}{\beta} \int_T^0 \exp\left(\frac{-E_a}{RT}\right) \cdot dT \]  

The E_a for thermal decomposition evaluated using Friedman isoconversional models [23,24]. By plotting a standard straight-line equation y = mx + c by referring to , the E_a can be obtained from the slope of each model equation at different conversion factor (α = 0.1 – 0.9). Friedman method is known as differential and most straightforward method to determine E_a using equation (6) [24–26]. The

\[ \frac{da}{dt} = - \frac{dw}{wi-wf}, \]  

where the \( \frac{dw}{dt} \) is the thermal degradation rate obtained from DTG curve [27].

\[ \ln \beta \frac{da}{dt} = - \frac{E_a}{RT} + \ln A + \ln f(\alpha) \]
3. Results and Discussion

3.1. CPO and TPS effect on thermal and mechanical properties of PE/TPS/CPO film

3.1.1. Thermal degradation. Thermal degradation by weight loss of PE/TPS/CPO films is illustrated in the TG curve (Figure 1) and the rate of weight loss in the derivative thermogravimetric (DTG) curve (Figure 2). Based on Figure 1, the thermal degradation profile is generally divided into three main stages. At stage 1, the evaporation of water occurred at around 90 °C representing the presence of physiosorbed water (moisture trapped) during the film preparation [28]. This temperature shows the evaporation of moisture content and the volatile compound [29]. At stage 2, the peak temperature occurred was 305.94 °C (Film-1) and 305.71 °C (Film-2) for 1g CPO, while 306.09 °C (Film-3) and 306.28 °C (Film-4) for 4.5g CPO. This temperature degradation represents glycerol-starch evaporation. It shows the interaction between glycerol and starch in the film occurred where the glycerol evaporation is commonly at 180 °C while starch decomposes at 340 °C [30]. More CPO has shifted (increased) the peak temperature by improving the interaction between glycerol and starch [31]. At this stage, the percentage of weight loss was around 9.56 % (Film-1) and 8.95 % (Film-3) with 6g TPS. The film with a higher TPS amount (15g), having a higher mass loss percentage of 16.9 % (Film-2) and 13.1 % (Film-4). At constant TPS, with a higher amount of CPO, the weight loss was reduced. It indicates that CPO enhances the thermal stability of films. Kamarudin [32] found similar thermal stability results improved with 3 % CPO into PP from 459.75 °C to 466.83 °C.

Finally, at stage 3, the PE started to degrade at 476 – 479 °C and a stable curve of degradation up to 500 °C. Others found PE began to degrade at 450 °C [33], while Nguyen [30] obtained linear low-density PE degrade at 491 °C. Figure 2 shows the degradation rate at each stage. The degradation rate of glycerol-starch was 0.49 mg/min (Film-1), 0.85 mg/min (Film-2), 0.39 mg/min (Film-3) and 0.66 mg/min (Film-4) respectively. The presence of more starch had increased the degradation rate of glycerol-starch. However, inconsistent PE degradation rate is shown by Film-4 with 2.08 mg/min, where the other films obtained almost the same degradation rate value (3.55 mg/min for Film-1, 3.83 mg/min for Film-2, and 3.95 mg/min for Film-3). Overall, TPS reduced the film's thermal stability while CPO only provides small changes in its thermal stability.

3.1.2. Thermal properties. Table 2 tabulated the data analyzed from Figure 3 and Figure 4. Figure 3 shows the DSC heating curve (second heating cycle) that gives the results of T_m and ΔH_m. Figure 4 displays the curve obtained from the cooling process during DSC analysis. The cooling curve provides crystallization behaviour of the polymer by giving the T_c and ΔH_c. Only one endothermic peak (Figure 3) and one exothermic peak (Figure 4) appeared in the DSC scan. Based on Table 2, there are no
significant changes in $T_m$, the value between 103.13 °C and 103.93 °C was obtained. Same goes to $T_c$, only 1.61 °C changes occurred with a minimum temperature of 90.42 °C and a maximum of 92.03 °C. $\Delta H_m$ and $\Delta H_c$ gives a significant change at a different percentage of TPS but only small changes at other CPO content. At constant CPO, the higher TPS gives a lower value of $\Delta H_m$ and $\Delta H_c$. Film-1 and Film-2 has less $\Delta H_m$ by 15 % with the addition of 15g TPS and for Film-3 and Film-4 (at constant 4.5g CPO), 24 % lesser $\Delta H_m$ when 15g TPS is added into the film. The high value of $\Delta H_m$ showing the ability of the polymer to withstand the heat and be more relax. The addition of more starch had disturbed the polymer relaxation and caused steric hindrance at once restricted PE movement. Panrong [34] also suggested incorporating modified starch into PE had increased the hydrophobic nature of PE and reduced polymer relaxation. For $\Delta H_c$, more TPS also has reduced the value by at least 14.45 J/g at 1g CPO (Film-1 and Film-2) and 6.22 J/g at 4.5g CPO (Film-3 and Film-4). The dispersion of more TPS into PE film caused a reduction in motion to form interface bonding at once preventing the arrangement of more crystalline phases [30]. A similar result obtained by Hammache [35] where $\Delta H_c$ reduced with more starch added into polypropylene film. The degree of crystallinity increased with more CPO content. Focusing on 6g TPS (Film-1 and Film-3), more CPO amount had increased the $X_c$ by 21.7 %, from 14.27 % to 18.23 % due to CPO's lubricating effect towards PE/TPS [32].

![Figure 3: DSC heating curve](image1)

![Figure 4: DSC cooling curve](image2)

**Table 2.** Thermal properties data from DSC cooling and heating curve

| Sample Name       | $T_m$ (°C) | $\Delta H_m$ (J/g) | $T_c$ (°C) | $\Delta H_c$(J/g) | $X_c$ (%) |
|-------------------|------------|--------------------|------------|-------------------|-----------|
| Film-1 (30PE/6TPS/1CPO) | 103.52    | 33.91              | 92.03      | 56.51             | 14.27     |
| Film-2 (30PE/15TPS/1CPO) | 103.93    | 28.76              | 91.53      | 42.06             | 15.05     |
| Film-3 (30PE/6TPS/4.5CPO) | 103.13    | 39.57              | 90.42      | 55.55             | 18.23     |
| Film-4 (30PE/15TPS/4.5CPO) | 103.18    | 29.86              | 90.99      | 49.33             | 16.82     |

3.1.3. Mechanical properties. Figure 5 displays the mechanical properties of the film (tensile strength (TS) and elongation at break (EAB). It shows that more starch and CPO directly reduced the TS and EAB of PE film. At constant PE/TPS, more CPO reduced TS by 28.27 % and EAB by 61.3 % for Film-3 compared to Film-1, while for Film-4, compared to Film-2, the TS reduced by 34.75 % and EAB reduced by 42.21 %. At constant PE/CPO, either 1g CPO or 4.5g CPO, the addition of more starch reduced TS by 8.88 % to 17.11%, and EAB reduced by 14.98 % to 43.06 %. A similar decreasing TS and EAB trend was also obtained when more starch was added into PE because starch increases of PE's stiffness correspond to decrease TS and EAB [15]. A study Ratnam [16] shows similar results where EAB falls as CPO's addition in LLDPE and HDPE increases. This situation could be due to the plasticization effect in the LDPE/TPS/CPO films. However, studies by Jusoh [17,18] obtained contradictory results on EAB. The values of EAB increase as CPO content increases. This case may be
due to the resins not incorporated with starch; hence, they have lower stiffness than resins combined with starch [19].

| Tensile strength (Mpa) | Elongation at break (%) |
|-----------------------|-------------------------|
| 6.19                  | 23.8                    |
| 5.64                  | 13.55                   |
| 4.44                  | 9.21                    |
| 3.68                  | 7.83                    |

**Figure 5:** Mechanical properties of the film at a different formulation

3.2. Heating rate effects on the thermal degradation and kinetic analysis of PE/TPS/CPO

3.2.1. Thermal degradation. Figure 6 shows the results of TG and DTG of pyrolysis of the PE/TPS/CPO film at different heating rates 7.5 °C/min (first heating rate, (1st-HR)), 10 °C/min (second heating rate, (2nd-HR)) and 12.5 °C/min (third heating rate (3rd-HR)). The decomposition stages were the same as in figure 1, the moisture content evaporation between 101 – 115 °C for 1st-HR, 105.73 – 123 °C for 2nd-HR, and 105 – 124 °C for 3rd-HR. Then, the glycerol-starch decomposed thermally between 265 – 298 °C, 273 – 318 °C, 276 – 325 °C. Finally, the PE/CPO deteriorated at 438 – 492 °C, 444 – 498 °C, 454 – 502 °C at increasing heating rate and some residue left. The sample's significant decomposition was completed below 500 °C, and there is no weight loss occurred anymore with the residue left was 5.06 mg for 1st-HR, 7.86 mg for 2nd-HR, and 4.22 mg for 3rd-HR. As shown in the DTG curves of figure 6, glycerol-starch and PE/CPO thermal decomposition rates increased when the heating rate increased. Peak temperature and degradation rate for glycerol-starch show an increasing trend. The peak temperature was 295.90 °C, 301.79 °C, and 305.98 °C, while the degradation rate was 0.51, 0.54, 0.88 mg/min respectively for 1st-HR, 2nd-HR, and 3rd-HR. The same situation occurred for peak temperature and degradation rate of PE/CPO decomposition (471.55 °C, 478.01 °C, and 478.86 °C; 2.22, 2.44, 3.87 mg/min). A similar trend observed by Kumar [25], the rate of banana leaves degradation increased with increasing heating rate from 297 °C to 316 °C. This condition is also supported by Mishra [19]. At a lower heating rate, lower peak temperature and degradation rate obtained suggest that better heat transfer occurred. A higher heating rate decreased heat distribution, which was also supported by [36].

**Figure 6:** TG and DTG curves for the pyrolysis of PE/TPS/CPO film at different heating rates
3.2.2. **Kinetics analysis using different kinetics model equations.** In this paper, the Friedman method employed to determine the $E_a$ of the film. Figure 7 represents the slopes at different conversion factor ($\alpha$), 0.1 to 0.9, calculated and plotted based on Friedman method for Film-5 that consist of PE/TPS/CPO. The activation energy ($E_a$) depicting the minimum energy needed to cause a chemical reaction to exist, and higher values demonstrate a slower reaction occurrence. At each conversion factor, a linear line obtained, and the slope represents the value for calculating the $E_a$ based on equation (6) and been tabulated in Table 3. Table 3 listed the $E_a$ and $R^2$ obtained with most of the $\alpha$ has a correlation coefficient of more than 0.9, indicates that the experimental data at the best-fitted value. From Table 3, the average $E_a$ obtained was 364.68 kJ/mol with the maximum $E_a$ was 1608 kJ/mol obtained at $\alpha$ equal to 0.2 while the minimum was at $\alpha$ equal to 0.3 with 107.87 kJ/mol. The average $E_a$ reported for PE decomposition was 146 kJ/mol based on the Kissinger method [37]. Ziming [38] found the $E_a$ from the thermal degradation of PE was about 121 kJ/mol to 141 kJ/mol at the different heating rate in K/min, while 126.09-275.81 kJ/mol depending on the heating rate and type of kinetic models used [39]. Curves for $E_a$ as a function of conversion factor was demonstrated in figure 8 and it shows the curves between $E_a$ and $\alpha$ was not linear. Friedman method is an effective method to compute $E_a$ that varies depending on $\alpha$ value. Plus, related to a simple differential form of kinetic rate law [19,23]. The $E_a$ expanded from $\alpha$, 0.1 to 0.2, and incredibly diminished from 0.2 to 0.3. Starting at 0.3, the $E_a$ expanded slowly when the change increment from 0.3 to 0.9, indicating the endothermic cycle happened while diminishing $E_a$ speaking to the exothermic cycle [25]. Higher values of $E_a$ showed the improvement in the thermal stability of the film [23].

![Figure 7: Regression lines to PE/TPS/CPO film based on Friedman method at 7.5, 10.0, and 12.5 °C/min](image)

| Conversion factor | slope     | $E_a$ (kJ/mol) | $R^2$  |
|-------------------|-----------|---------------|--------|
| 0.1               | -17924    | 149.02        | 0.9965 |
| 0.2               | -193409   | 1608.00       | 0.9964 |
| 0.3               | -12975    | 107.87        | 0.7073 |
| 0.4               | -22782    | 189.41        | 0.9997 |
| 0.5               | -27539    | 228.96        | 0.9998 |
| 0.6               | -30460    | 253.24        | 0.9945 |
| 0.7               | -29193    | 242.71        | 0.9898 |
| 0.8               | -29952    | 249.02        | 0.999  |
| 0.9               | -30536    | 253.88        | 1      |
4. Conclusion

Three steps of weight loss occurred in the film's thermal degradation representing the degradation of moisture, glycerol-starch, and CPO-PE degradation with significant weight loss by CPO-PE at onset temperature 401 °C and endset temperature at 524 °C. Reduction in enthalpy of fusion ($\Delta H_m$) was due to the presence of TPS that also reduces the degree of crystallinity of the film. However, CPO in PE/TPS enhanced the film's degree of crystallinity by giving a lubricating effect. Sadly, the tensile strength and elongation at break did not improve with the addition of CPO. The study continues with heating rates' impact on PE/TPS/CPO film. At a lower heating rate, better heat transfer occurred at once lower peak temperatures and lower degradation rates. By using Friedman method, the average activation energy obtained was 364.68 kJ/mol. The slowest reaction occurred at a 0.2 conversion factor, and the highest reaction rate was at a 0.3 conversion factor. Further comparison of kinetics should be conducted using other isoconversional methods such as Flynn-Wall-Ozawa, Kissinger, and modified Coats-Redfern to determine the activation energy's reliability obtained.

5. References

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