Co-Pyrolysis of Empty Fruit Bunch and High-Density Polyethylene Over HZSM-5: Thermogravimetric, Kinetic and Thermodynamic Analysis

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ABSTRACT

This study conducted non-catalytic and catalytic co-pyrolysis of empty fruit bunch (EFB) and high-density polyethylene (HDPE) with HDPE-to-EFB mass ratios of 1:0, 0:1, and 1:1 via thermogravimetric analyser (TGA) and the application of Coats-Redfern method for kinetic and thermodynamic analysis. Hydrogen-exchanged zeolite socony mobil-5 (HZSM-5) catalyst was used with a catalyst-to-feedstock mass ratio of 1:1 for all the catalytic samples. From TGA results, the highest amount of volatilized matter in Phase II was obtained from non-catalytic pyrolysis of HDPE (NCP: 98.6 wt%) while the lowest amount of volatilized matter in Phase II was obtained from non-catalytic pyrolysis of EFB (NCB: 67.3 wt%). The activation energy for the pyrolysis of HDPE was highest followed by the co-pyrolysis of EFB and HDPE and pyrolysis of EFB, for both non-catalytic and catalytic runs. The activation energy based on the HDPE-to-EFB mass ratio was obtained in the following order: NCP (353.6 kJ/mol) > CP (214.3 kJ/mol) > NCPB (109.6 kJ/mol) > CPB (64.7 kJ/mol) > NCB (25.8 kJ/mol) > CB (24.4 kJ/mol). For thermodynamic analysis, ΔH and ΔG were positive for all the runs while ΔS, was negative for the non-catalytic and catalytic pyrolysis of EFB and co-pyrolysis of HDPE and EFB (NCB, NCPB, CB and CPB) and positive for the non-catalytic and catalytic pyrolysis of HDPE (NCP and CP).

Keywords: pyrolysis kinetics, pyrolysis thermodynamics, co-pyrolysis, biomass, plastic, HZSM-5

NONMENCLATURE

Abbreviations

CB	Catalytic pyrolysis of EFB over HZSM-5
CP	Catalytic pyrolysis of HDPE over HZSM-5
CPB	Catalytic co-pyrolysis of EFB and HDPE over HZSM-5
CR	Coats-Redfern
EFB	Empty fruit bunch
HDPE	High-density polyethylene
HZSM-5	Hydrogen-exchanged zeolite socony mobil-5
NCB	Non-catalytic pyrolysis of EFB
NCP	Non-catalytic pyrolysis of HDPE
NCPB	Non-catalytic co-pyrolysis of EFB and HDPE
TGA	Thermogravimetric analyser

Symbols

E_A	Activation energy
K_B	Boltzmann constant
ΔH	Change of enthalpy
ΔS	Change of entropy
ΔG	Change of Gibbs free energy
α	Fractional conversion
β	Heating rate
h	Planck's constant
A	Pre-exponential factor
T	Temperature
R	Universal gas constant

1. INTRODUCTION

Global energy demand has been increasing over the past few decades and to meet this demand, fossil fuels have been a primary energy driver due to its cheap availability and high energy density [1]. However, the extensive exploration of fossil fuels has not only led to the destruction of wildlife habitat, but also has been a large contributor to the greenhouse gas emissions [2]. Fossil fuel resources are not infinite and have been decreasing since 2018, based on a statistical report by British Petroleum, and thus, finding alternatives, such as biofuel derived from biomass, can help to reduce our dependency on fossil fuels [3].

Biomass is a renewable resource that is abundant, especially in countries that practice agriculture. Apart from woody biomass, biomass can also come from agricultural residues like empty fruit bunch (EFB), sugarcane bagasse and wheat straw. In Southeast Asia, Malaysia is one of the top palm oil producers and approximately 70% of the palm oil production consists of wastes like EFB [4]. EFB can be used as feedstock for boiler but is often discarded as waste due to its high moisture content from sterilization. Therefore, by utilizing EFB, valuable compounds, like hydrocarbons, can be retrieved from this resource. To obtain biofuels in the form of bio-oil, biomass is broken down via pyrolysis where biomass is thermally degraded between 300 to 700 °C in the absence of oxygen [5]. In pyrolysis, biomass undergoes dehydration, followed by fragmentation and finally formation of pyrolysis products like bio-oil, char and gas [6]. However, bio-oil derived from biomass is often obtained in low yield and low quality due to the presence of oxygenated compounds in the resulting bio-oil [7]. Therefore, biomass is paired with plastic waste, helping with the decomposition of biomass.

Plastic waste is a current global issue due to its mass production and unresolved disposal method. Among the different types of plastic, high-density polyethylene (HDPE) is one of the top types of plastic wastes produced in Malaysia [8]. Thus, utilizing plastic waste in pyrolysis of biomass will not only help in bio-oil production, but also in diverting waste from landfills and waterways. In the co-pyrolysis of biomass and plastic, hydrogen from plastic helps create a hydrogen atmosphere during pyrolysis, desired for the decomposition of biomass. Furthermore, the oxygen produced from the breakdown of biomass helps the cracking of plastic, creating a positive synergy that increases the bio-oil yield and quality [6]. For further improvement of bio-oil quality, catalysts are added to the biomass pyrolysis process, which further reduces oxygen content [5]. By adding catalysts, chemical reactions such as decarbonylation and decarboxylation are promoted, along with aromatization reactions, favouring the production of hydrocarbon content [9]. HZSM-5 is often reported to be the best catalyst for pyrolysis due to its microporous structure that favours aromatic hydrocarbons to pass through [10].

In addition to the pyrolysis process itself, the kinetic and thermodynamic aspects of pyrolysis are also important in understanding the thermal behaviour of the process. Kinetic parameters, such as Ea and A, and thermodynamic parameters, such as ΔH, ΔG, and ΔS, provide information that can be used for designing reactors at an industrial scale. These parameters are obtained using thermogravimetric analyser (TGA) and the application of integral methods, such as Coats-Redfern (CR) method. However, in the pyrolysis field, there are lack of studies on the direct comparison on the kinetic and thermodynamic analysis of pyrolysis and co-pyrolysis, with and without catalyst. Therefore, in this study, kinetic and thermodynamic analysis were conducted on the pyrolysis of EFB, pyrolysis of HDPE and the co-pyrolysis of EFB and HDPE with and without HZSM-5 using TGA and the application of CR method. TGA is commonly used as a pyrolyser to investigate the thermal degradation behavior of material and its kinetic parameters. Since TGA is not equipped with analytical instruments like gas chromatography–mass spectrometry (GC/MS), the limitation is that chemical components obtained from pyrolysis were not reported in this study. Thus, the focus of this study is on the degradation behavior, kinetic and thermodynamics of pyrolysis.

2. MATERIALS AND METHODS

2.1 Biomass and plastic feedstock

Empty fruit bunch (EFB) was selected as the biomass feedstock and high-density polyethylene (HDPE) as the plastic feedstock. EFB and HDPE were purchased separately from a local supplier in Malaysia. The preparation of biomass sample can be referred to in our previous work [11]. HDPE samples were ground in a lab blender into fine particles and sieved to obtain a particle size of 0.50 mm.

2.2 Catalyst preparation and characterization

Zeolite ZSM-5 (Si/Al = 23) was purchased from Alfa Aesar. The preparation and results of the

physicochemical properties of HZSM-5 catalyst can be referred to in our previous work [11].

2.3 Experimental setup

Non-catalytic and catalytic pyrolysis runs were conducted using a thermogravimetric analyser (TGA/SDTA851, METTLER TOLEDO, USA) according to American Society for Testing and Materials (ASTM D-3172). For non-catalytic runs, the samples were prepared following a HDPE-to-EFB mass ratio of 1:0, 0:1 and 1:1, and labelled as NCP, NCB, and NCPB, respectively. For the catalytic runs, a fixed catalyst-to-feedstock mass ratio of 1:1 was prepared for the samples following a HDPE-to-EFB mass ratio of 1:0, 0:1 and 1:1, and labelled as CP, CB, and CPB, respectively. All samples were prepared using a mortar and pestle to get a uniform mixture. Procedure for TGA pyrolysis for catalytic and non-catalytic samples can be referred to in our previous work with only change of heating rate at 20°C/min [7].

3. THEORY

Coats-Redfern (CR) method was applied to obtain kinetic parameters, such as E_a and A. The first-order reaction model was used as it simplifies the pyrolysis reaction into a one-step process as shown in Eq. (1) [12],

$$Feedstock \rightarrow Volatiles + Char \qquad (1)$$

where feedstock, for this study, is either, biomass only, plastic only or biomass and plastic. The derivation of the CR method can be referred to in our previous work [7]. With the application of the first-order reaction model, the resulting linearized expression that will be used is shown in Eq. (2),

$$\ln\left[\frac{(1-\alpha)^{-1}}{T^2}\right] = \ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \qquad (2)$$

where α is the fractional conversion of the sample, T is the temperature in Kelvin, and R is the universal gas constant (8.3144 J/mol·K), and β is the heating rate. An xy-plot can be generated from the TGA data following Eq. (2), and E_a and A can be determined.

From E_a and A, the thermodynamic values, such as ΔH, ΔG, and ΔS, can be calculated respectively. Eq. (3) – (5) shown below were applied from Chen et al. (2021) [13].

$$\Delta H = E_A - RT \qquad (3)$$

$$\Delta G = E_A + RT \ln\left(\frac{K_B T_m}{hA}\right) \qquad (4)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T_m} \qquad (5)$$

where K_B is the Boltzman constant (1.381 × 10^{-23} J/K), h is Planck's constant (6.626 × 10^{-34} Js) and T_m is the temperature corresponding to the highest peak in the DTG curve (K).

4. RESULTS AND DISCUSSION

4.1 Thermogravimetric analysis

Fig. 1 and 2 shows the resulting thermogravimetric (TG) and derivative thermogravimetric (DTG) curves respectively for the individual pyrolysis of EFB and HDPE, the pyrolysis of the feedstock blend and the results for the catalytic runs. The TG and DTG curves were divided into three phases, where Phase I (30 to 200°C) represents the vaporization of moisture and light components, Phase II (200 to 520°C) represents the volatilization of biomass, specifically hemicellulose and cellulose, and plastic and Phase III (520 to 700°C) represents lignin degradation [7]. From TG curves, the degradation pattern change affected by the type of feedstock and the presence of catalyst was observed. In Fig. 1, for the non-catalytic samples (NCP, NCB, and NCPB), EFB starts to degrade at a lower temperature of 200°C compared to HDPE, which starts to degrade at 400°C. This is due to the structures of the feedstock where EFB, having an amorphous structure, requires lower temperature to degrade compared to HDPE, which has a crystalline structure [14]. When feedstock was mixed together, an overlapping of the TG curves was observed. From DTG curves, the rate of mass loss of volatilized matter for each run can be seen. In Fig. 2, for the non-catalytic runs, the rate of mass loss for pyrolysis of EFB (NCB) was high within the range of 200 to 370 °C as seen from the peaks in the DTG curves. This is due to the lignocellulosic components, specifically cellulose and hemicellulose, that thermally degrades within the mentioned temperature range [14]. For non-catalytic pyrolysis of HDPE (NCP), the rate of mass loss was highest around 490°C, where a single peak was observed in Fig. 2. Similar to its TG curve, when feedstock was mixed together, the overlapping of the DTG curves was seen where peaks were observed in the 200 to 370°C and the 400 to 520°C.

Similar degradation patterns were seen, in Fig. 1 and Fig. 2, for the catalytic runs (CP and CB) where, EFB degrades at a lower temperature than HDPE, even with the presence of catalyst. This also occurred in catalytic co-pyrolysis (CPB) where its degradation pattern lies between that of CP and CB, and was also seen for the non-catalytic case (NCPB). The DTG curves for the catalytic runs, in Fig. 2, show ranges where peaks are

present that are similar to its non-catalytic runs with differences in the intensities of the peaks. The difference in the intensities implies that the presence of catalysts can affect the rate of decomposition of feedstock by increasing or slightly suppressing the rate of decomposition during pyrolysis.
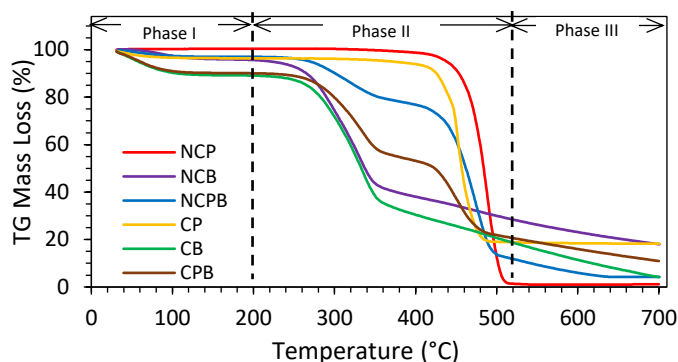

Fig. 1. TG curves for non-catalytic and catalytic pyrolysis samples
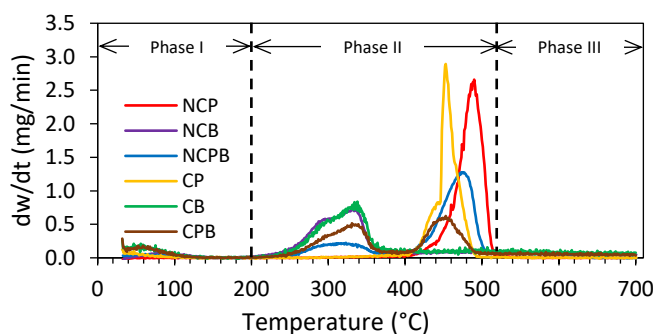

Fig. 2. DTG curves for non-catalytic and catalytic pyrolysis samples

Fig. 3 shows the breakdown of volatilized matter for the non-catalytic and catalytic pyrolysis runs. For pyrolysis of HDPE, when catalysts were added, mass loss in Phase II reduced from 98.6 wt% (NCP) to 77.5 wt% (CP), while solid residual left increased from 1.1 wt% to 18.3 wt%. The solid residual increase was attributed to the coke formed from the melting of plastic into wax and the further reaction of the wax molecules on the acid sites of the catalyst [15]. For pyrolysis of EFB, with the addition of catalysts, mass loss in Phase II increased from 67.3 wt% (NCB) to 70.4 wt%

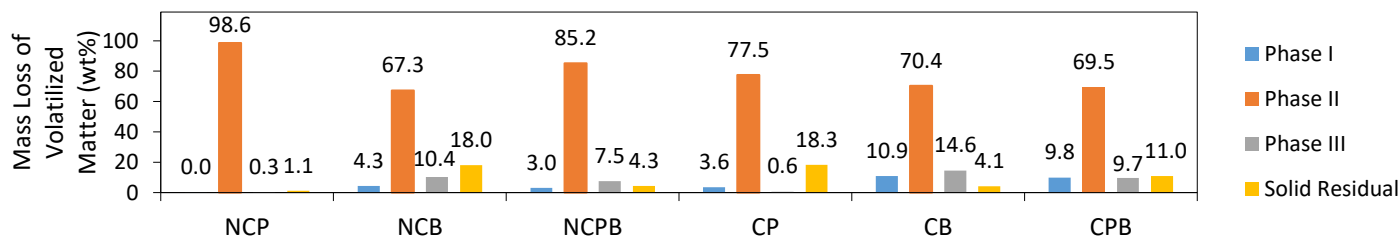(CB), while solid residual left decreased from 18.0 wt% to 4.1 wt%. This is due to the additional reaction sites provided by the catalyst allowing more matter to be volatilized. For the co-pyrolysis of HDPE and EFB, the presence of catalyst promoted the production of coke where volatilized matter in Phase II was reduced from 85.2 wt% (NCPB) to 69.5 wt % (CPB), and solid residual increased from 4.3 wt% to 11.0 wt%. Iftikhar et al. (2019) also observed a reduction in liquid yield for the catalytic co-pyrolysis of sugarcane bagasse and polystyrene over HZSM-5 but the liquid yield of the catalytic run was reported to have higher hydrocarbon content [16]. Thus, further analysis on the volatilized matter of this study should be done to determine the hydrocarbon content.

4.2 Kinetic analysis

Table 1 shows E_a and A, determined from the collected TGA data and the application of CR method. Moreover, to observe how strongly the first-order reaction model was related to the pyrolysis reaction, R^2 value for each of the runs was included in Table 1. Based on Table 1, almost all the runs had an R^2 value close to or above 0.90, indicating that the pyrolysis reaction strongly models the first-order reaction. An exception was seen for the catalytic co-pyrolysis of HDPE and EFB (CPB) where the R^2 value was 0.810. This could imply that the presence of catalysts with the mixing of feedstock could lead the deviation of the pyrolysis reaction from the first-order reaction model. Without the presence of catalysts, pyrolysis of HDPE (NCP) had a very high E_a (353.6 kJ/mol) compared to pyrolysis of EFB (NCB), which was 25.8 kJ/mol. E_a represents the energy barrier that needs to be overcome by the reaction for products to form. Since HDPE has a more crystalline structure compared to EFB, this energy barrier for pyrolysis of HDPE is much higher than that of EFB. Furthermore, the mixing of feedstock of HDPE and EFB has successfully reduced E_a to 109.6 kJ/mol (NCPB). The catalytic runs show that with the presence of catalysts, the E_a can be further reduced, as seen in Table 1, where overall, the values for E_a for the

Fig. 3. Volatile mass loss for non-catalytic and catalytic samples

catalytic runs were lower than their counter non-catalytic runs. The presence of catalysts helps to increase reaction activity during pyrolysis, which reduces the energy barrier for reactions to occur, resulting in the reduction of E_a [12]. Pre-exponential factor, A, describes the number of collisions between molecules during the pyrolysis reaction. For both non-catalytic and catalytic runs, the pyrolysis of HDPE had higher A compared to the pyrolysis of EFB and with the mixing of feedstock, the reduction of A value was observed.

Table 1. Kinetic parameters obtained from TGA pyrolysis runs

Run	E_a (kJ/mol)	A (min^{-1})	Average R^2
NCP	353.6	8.5×10^{25}	0.884
NCB	25.8	40.7	0.921
NCPB	109.6	2.2×10^8	0.947
CP	214.3	6.4×10^{16}	0.937
CB	24.4	36.8	0.934
CPB	64.7	4.5×10^4	0.810

4.3 Thermodynamic analysis

Table 2 shows the thermodynamic parameters, ΔH, ΔG and ΔS, calculated from E_a and A. ΔH quantifies how much heat has been absorbed or released during pyrolysis. The values for ΔH for all the pyrolysis runs in Table 2 are positive, indicating an endothermic process, where heat was absorbed during pyrolysis. Pyrolysis runs for HDPE, for both catalytic and non-catalytic cases, had high ΔH, 347.3 kJ/mol (NCP) and 208.3 kJ/mol (CP) respectively, compared to non-catalytic and catalytic pyrolysis of EFB (NCB and CB). This is due to the crystalline structure of HDPE which requires more energy, hence more heat to break down the structure compared to EFB. ΔG represents how much energy available in a system that is able to do work. When HDPE is added to EFB, ΔG increases for both cases of non-catalytic and catalytic, 204.6 kJ/mol (NCPB) and 208.0 kJ/mol (CPB), respectively. This indicated that mixing feedstock results in more available energy to break down the feedstock during the pyrolysis process. In addition, the positive values of ΔG for all of the runs indicated that the pyrolysis process is non-spontaneous, as heat is applied for pyrolysis to happen. ΔS represents the degree of disorder in a system where a positive ΔS indicates more disorder while a negative ΔS indicates less disorder. It was observed that for non-catalytic and catalytic pyrolysis of HDPE (NCP and CP), positive ΔS values were obtained, indicating that the system

becomes more disordered during the pyrolysis reaction. This also implies that the system has high reactivity with less reaction time, resulting in more volatilization of components [17]. For pyrolysis of biomass and co-pyrolysis (NCB, NCPB, CB and CPB), negative ΔS values were obtained, implying that the biomass feedstock or the mixed feedstock system becomes less disordered during the pyrolysis reaction. This resulted in the low reactivity of the system with less volatilization of components compared to the pyrolysis of HDPE.

Table 2. Thermodynamic parameters for all pyrolysis runs

Run	ΔH (kJ/mol)	ΔG (kJ/mol)	ΔS (kJ/mol·K)
NCP	347.3	193.6	0.201
NCB	20.8	180.2	-0.262
NCPB	103.3	204.6	-0.135
CP	208.3	188.6	0.027
CB	19.3	180.2	-0.263
CPB	58.7	208.0	-0.206

5. CONCLUSION

In this study, the thermogravimetric, kinetic, and thermodynamic analysis was successfully conducted on pyrolysis of EFB and HDPE individually and the co-pyrolysis of EFB and HDPE with and without HZSM-5 catalyst via TGA and the application of CR method. From TG curves, the mixing of EFB and HDPE feedstock resulted in the overlapping of TG curves of individual feedstock pyrolysis. Non-catalytic pyrolysis of HDPE (NCP) resulted in the highest volatilized matter loss in Phase II (98.6 wt%) while non-catalytic pyrolysis of EFB (NCB) resulted in the lowest volatilized matter loss in Phase II (67.3 wt%). Regarding kinetic parameters, the highest E_a value of 353.6 kJ/mol was obtained from the non-catalytic pyrolysis of HDPE (NCP) while the lowest E_a value of 24.4 kJ/mol was obtained from the catalytic pyrolysis of EFB (CB). Positive values of ΔH and ΔG were obtained for all the pyrolysis runs, with the highest ΔH value obtained from non-catalytic pyrolysis of HDPE (347.3 kJ/mol) and the highest ΔG value obtained from catalytic co-pyrolysis of EFB and HDPE (208.0 kJ/mol). For ΔS, negative values were obtained for the non-catalytic and catalytic pyrolysis of EFB and co-pyrolysis of HDPE and EFB (NCB, NCPB, CB and CPB) while positive values were obtained for the non-catalytic and catalytic pyrolysis of HDPE (NCP and CP), where the highest ΔS value was obtained from the non-catalytic pyrolysis of HDPE (NCP, 0.201 kJ/mol·K). From this

study, it can be seen that the addition of HDPE to EFB in non-catalytic pyrolysis can increase amount of volatilized matter in Phase II while adding catalysts to the co-pyrolysis process can help to decrease E_a. These results can be considered for future work via pyrolyser-GC/MS in analysing the chemical components produced from the co-pyrolysis of EFB and HDPE over HZSM-5.

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