

# Synergic Effect and Kinetic Mechanisms for Co-Pyrolysis of Empty Fruit Bunch and Palm Oil Sludge

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## ABSTRACT

The thermochemical behaviour of Co-Pyrolysis between Empty Fruit Bunches (EFB) and Palm Oil Mill Effluent (POME) sludge were studied using Thermogravimetric Analysis (TGA). EFB, POME sludge, and their blends (EFB : POME sludge of 100:0, 90:10, 75:25, 50:50, 25:75, and 0:100) were studied at different heating rates (5, 10, 20, 30, and 40°C/min) with nitrogen (N<sub>2</sub>) purge of 20 ml/min to simulate pyrolysis conditions. All the samples experienced three pyrolysis stages and for each stage, the mechanisms responsible were determined. During co-pyrolysis, a positive synergistic effect was observed in the experiments, which is a favourable finding. With the increase in POME sludge percentage in the blends, the activation energy for the main decomposition stage (Stage 2) decreased from 89.33 kJ/mol to 63.47 kJ/mol, when the kinetic model was first order reaction (F1).

**Keywords:** Co-Pyrolysis, EFB, TGA, POME Sludge, Synergistic Effect.

## 1.0 INTRODUCTION

Global energy demand has been on the rise and carbon dioxide (CO<sub>2</sub>) emissions are commonly associated with energy production, which contributes to climate change. Concerns on the effects of fossil fuel emissions on the environment and energy security have been raised. Among renewable energy sources, biomass shows high potential in terms of feed flexibility and abundance. Malaysia is known as the largest exporter of palm oil, and so, large amount of palm waste are being produced simultaneously. A review on the utilization of the palm waste in the production of biomass energy was conducted [1].

Liquid fuel is the energy source that is consumed most in the world. Thus, finding a potential substitute for liquid fuels is important. This study takes interest in fast pyrolysis, a thermochemical conversion method of biomass to obtain liquid fuel, known as bio-oil, due to the high oil yield and high energy density compared to virgin biomass. Abdullah *et al.*, characterized the bio-oil derived from EFB and obtained a low pH of between 2 and 3 [2]. Having a high acidity causes the bio-oil to be corrosive and not suitable to be directly used as direct fuel. Bio-oil derived from POME sludge has a high pH of about 9.4, signifying that the bio-oil is alkaline in nature. Thus, Thangalazhy-Gopakumar *et al.*, suggested the co-pyrolysis of lignocellulosic biomass and sludge in order to achieve a neutralized bio-oil [3].

Co-pyrolysis of biomass is one of the potential options to improve the quality of bio-fuel. Due to the differences in chemical and physical properties of different biomass, a different thermal reactivity may occur during co-pyrolysis [4].

A positive synergistic effect was observed in the co-pyrolysis of oil-palm solid wastes and paper sludge, contributing to an increase in both gas and liquid products, but decrease in char production [5]. This effect was favourable in achieving a higher liquid yield. However, co-pyrolysis between sewage sludge and pine sawdust did not show significant synergistic effect upon thermal degradation; an inhibitive effect was observed by Mu *et al.*, in the co-pyrolysis of petrochemical wastewater sludge with lignite, indicated by a negative synergistic effect [4], [6].

Biomass exhibits different behaviours in thermal process. The characterisation of EFB and POME have been done individually. However, the study on the reactivity and kinetics of the co-pyrolysis of EFB and POME have yet to be done. This study aims to investigate the thermal behaviour of the co-pyrolysis of EFB and POME upon fast pyrolysis.

## 2.0 MATERIALS AND METHODS

### 2.1 Feedstock Characterization

The feedstocks used in this work were palm Empty Fruit Bunches (EFB) and treated Palm Oil Mill Effluent (POME) sludge. The samples were collected from Seri Ulu Langat Palm Oil Mill Sdn. Bhd, Dengkil, Selangor (Malaysia). EFB was dried in an oven at 75°C for 16 h. Dried sludge, which was ready to compost in soil was collected and further sun dried for 3 days. The samples were then grounded to less than 2 mm in particle size. The feedstock properties are presented in Table 1. The moisture content was

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calculated as weight differences after being placed in the oven at 103°C for 16 hours. The ash content of the sample was determined per ASTM E 1755 standard. Higher heating values (HHV) of both EFB and POME sludge were measured using Parr 6100 bomb calorimeter. Different blends at different EFB to POME sludge ratios were prepared by mixing (100:0 90:10, 75:25, 50:50, 25:75, and 0:100).

**Table 1: Ultimate and Proximate Analyses and HHV of EFB and POME Sludge**

SAMPLE	PROXIMATE ANALYSIS		HHV (MJ/kg)
	Ash (%)	Moisture (%)	
EFB	1.42±0.17	5.10±0.77	18.24±0.11
POME	24.17±0.28	13.60±0.12	14.52±0.01

## 2.2 Mathematic Background

Thermogravimetric analysis was carried out in programmable TGA DSC 1 Mettler Toledo to examine the decomposition behaviour of biomass upon pyrolysis. The different EFB to POME sludge ratios were taken for co-pyrolysis studies. The samples were heated at a heating rate of 5, 10, 20, 30 & 40°C / min with nitrogen (N<sub>2</sub>) purge of 20 ml/min. In each experimental run, approximately 10 mg of biomass was heated from room temperature to 900°C, and held at that temperature for 10 mins. The experiments were duplicated to ensure the reproducibility of results.

### 2.2.1 Interaction

To study the existence of interaction between the EFB and POME sludge blends upon pyrolysis, the theoretical values for TG and DTG curves of the blends were calculated. Eqn. (1) shows the calculation to obtain the values by adding the decomposition curves of each individual component:

$$W = x_{EFB} \cdot W_{EFB} + x_{sludge} \cdot W_{sludge} \quad (1)$$

where,  $W$  is the theoretical TG or DTG values of the blends;  $W_{EFB}$  and  $W_{sludge}$  are experimental TG and DTG values of EFB and POME, respectively;  $x_{EFB}$  and  $x_{sludge}$  are mass percentages of EFB and POME in the blends.

### 2.2.2 Kinetic Model

Kinetic analysis of EFB, POME sludge, and their blends were carried out to obtain the kinetic triplets (activation energy (E), apparent pre-exponential factor (A), kinetic model  $f(\alpha)$ ).

The conversion degree,  $\alpha$  has been defined as the mass fraction of decomposed solid:

$$\alpha = \frac{m_o - m}{m_o - m_f} \quad (2)$$

where  $m_o$ ,  $m$ , and  $m_f$  are the initial, instantaneous, and final masses of the solids respectively.

In non-isothermal experiments, the rate of solid degradation can be written as:

$$\frac{d\alpha}{dt} = kf(\alpha) \quad (3)$$

where  $k$  is the temperature-dependant rate constant and  $f(\alpha)$  is a function of conversion that varies according to the reaction

model. The reaction constant,  $k$  can be expressed by Arrhenius equation

$$k = A \exp\left(-\frac{E}{RT}\right) \quad (4)$$

where  $A$  is the pre-exponential factor,  $E$  is the activation energy,  $R$  is the gas constant, and  $T$  is the absolute temperature. Substituting Eqn. (4) into Eqn. (3) gives

$$\frac{d\alpha}{dt} = A \exp\left(-\frac{E}{RT}\right) \cdot f(\alpha) \quad (5)$$

Considering a constant heating rate of  $\beta = \frac{dT}{dt}$ , Eqn. (5) can be rearranged to

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(-\frac{E}{RT}\right) \cdot f(\alpha) \quad (6)$$

Coats-Redfern (CR) method employs the integral form of Eqn. (6), as presented in Eqn. (7).

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_{T_0}^T \exp\left(-\frac{E}{RT}\right) dT \quad (7)$$

where  $g(\alpha)$  is the integrated form of the conversion dependence function  $f(\alpha)$ . CR method is further integrated, yielding:

$$\ln \left[ \frac{g(\alpha)}{T^2} \right] = \ln \left[ \frac{AR}{\beta E} \left( 1 - \frac{2RT}{E} \right) \right] - \frac{E}{RT} \quad (8)$$

As the temperature range applied in the combustion of the samples, the value of  $\frac{2RT}{E}$  was far less than one, so Eq. (9) is obtained.

$$\ln \left[ \frac{g(\alpha)}{T^2} \right] = \ln \left( \frac{AR}{\beta E} \right) - \frac{E}{RT} \quad (9)$$

where,  $\ln \frac{AR}{\beta E}$  is essentially a constant value.

A straight line should be obtained from the plotting of  $\ln \left[ \frac{g(\alpha)}{T^2} \right]$  against  $\frac{1}{T}$  as they have a linear relationship. If the correct  $g(\alpha)$  is used, the straight line should have a high correlation coefficient of linear regression analysis. Then, the values of  $E$  and  $A$  can be derived from the slope  $-\frac{E}{R}$  and the intercept  $\ln \left( \frac{AR}{\beta E} \right)$ , respectively [7]. By identifying  $g(\alpha)$  that gives the highest correlation coefficient, the pyrolysis reaction of the samples can be associated with the appropriate mechanisms. Table 2 shows the basic model functions that were used in this kinetic study of solid-state reactions.

**Table 2: Expressions of functions  $g(\alpha)$  and their corresponding mechanisms (Adapted from Vlaev et al., 2008; Gil et al., 2010)**

NO.	SYMBOL	NAME OF FUNCTION	$g(\alpha)$	RATE- DETERMINING MECHANISM
<b>1. Chemical process or mechanism non-invoking equations</b>				
1.1	$F_{1/3}$	One-third order	$1 - (1-\alpha)^{2/3}$	Chemical reaction
1.2	$F_{3/4}$	Three-quarters order	$1 - (1-\alpha)^{1/4}$	Chemical reaction
1.3	$F_{3/2}$	One and a half order	$(1-\alpha)^{-1/2} - 1$	Chemical reaction
1.4	$F_1$	First Order	$-\ln(1-\alpha)$	Chemical reaction

1.5	$F_2$	Second Order	$(1 - \alpha)^{-1} - 1$	Chemical reaction
1.6	$F_3$	Third Order	$(1 - \alpha)^{-2} - 1$	Chemical reaction
2. Phase boundary reaction				
2.1	$R_1, F_0, P_1$	Power law	$\alpha$	Contracting disk
2.2	$R_2, F_{1/2}$	Power law	$1 - (1 - \alpha)^{1/2}$	Contracting cylinder
2.3	$R_3, F_{2/3}$	Power law	$1 - (1 - \alpha)^{1/3}$	Contracting sphere
3. Based on the diffusion mechanism				
3.1	$D_1$	Parabola law	$\alpha^2$	One-dimensional diffusion
3.2	$D_2$	Valensi equation	$\frac{\alpha + (1 - \alpha)}{\ln(1 - \alpha)}$	Two-dimensional diffusion
3.3	$D_3$	Jander equation	$[1 - (1 - \alpha)^{1/3}]^2$	Three-dimensional diffusion, spherical symmetry
3.4	$D_4$	Ginstling-Brounstein equation	$[1 - \frac{2\alpha}{3} - (1 - \alpha)^{2/3}]^2$	Three-dimensional diffusion, cylindrical symmetry
3.5	$D_5$	Zhuravlev, Lasokin, Tempelman equation	$[(1 - \alpha)^{-1/3} - 1]^2$	Three-dimensional diffusion
3.6	$D_6$	anti-Jander equation	$[(1 + \alpha)^{1/3} - 1]^2$	Three-dimensional diffusion
3.7	$D_7$	anti-Ginstling-Brounstein equation	$1 + \frac{2\alpha}{3} - (1 + \alpha)^{2/3}$	Three-dimensional diffusion
3.8	$D_8$	anti-Zhuravlev, Lasokin, Tempelman equation	$[(1 + \alpha)^{-1/3} - 1]^2$	Three-dimensional diffusion

obtained a positive synergic effect in the co-pyrolysis of legume straw and coal [12]. In that study, the biomass was considered as hydrogen donor, which aided the hydrogenation of coal upon pyrolysis, resulting in some positive synergies.

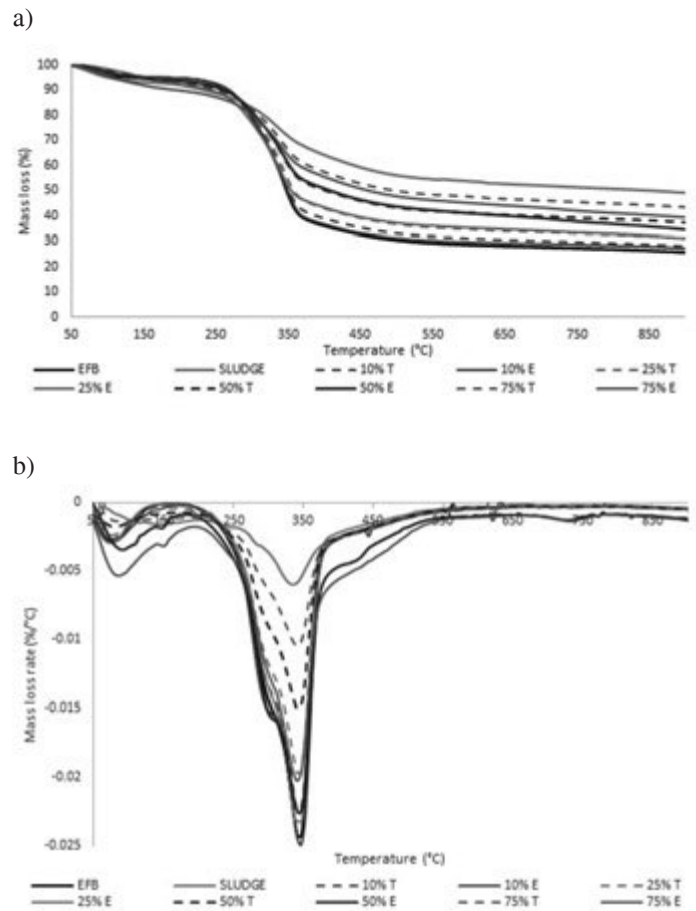


Figure 1: (a) TG curves and (b) DTG curves of EFB, POME sludge, and their blends at  $\beta=20^\circ\text{C}/\text{min}$ .

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Synergic Effect

Figure 1 depicts the TG and DTG curves of EFB, POME sludge, and their blends at a heating rate of  $20^\circ\text{C}/\text{min}$  in the presence of  $\text{N}_2$ . With the increase of sludge percentages in the blends, the profiles of both the TG and DTG curves shifted from EFB to sludge where combustion reactivity gradually decreased.

The understanding of synergic effect in the production of bio-oil via co-pyrolysis is important as it is one of the main factors responsible in the measuring or determining improvements in oil quality and quantity [10]. Positive or negative synergic effect relies on the type and contact between the components, duration of pyrolysis, temperature and heating rate, removal or equilibrium of volatiles formed, and addition of solvents, catalysts, and hydrogen donors [10].

In order to investigate the interaction between EFB and POME sludge, the theoretical and experimental values of blends during co-pyrolysis were compared to and presented in Figure 1. Experimental values showed higher mass loss as compared to those of theoretical values, suggesting that more cellulose and hemicellulose were being degraded than expected. This finding hinted the existence of interaction between EFB and POME sludge, known as the synergistic effect [11]. Zhang *et al.*,

#### 3.2 Kinetics and Mechanisms of Co-pyrolysis by Coats-Redfern (CR) Method

Using Coats and Redfern (CR) method, the solid-state mechanisms involved in the pyrolysis of EFB, POME sludge, and their blends were determined to guide and optimize production. Based on the mechanisms that gave the highest correlation coefficient ( $R^2$ ), the kinetic triplets (E, A, and  $f(x)$ ) of the stages were calculated and identified.

Based on TGA results as presented in Table 3, the samples were divided to three different stages and analysed separately. Stage 1 ranges from 40 to  $200^\circ\text{C}$ ; Stage 2 from 200 to  $400^\circ\text{C}$ ; and Stage 3 from 400 to  $650^\circ\text{C}$ . The highest correlations ranged between 0.9774 – 0.9992, indicating the reliability of the kinetic parameters obtained.

Table 3: Kinetic parameters for EFB, POME sludge, and their blends at a heating rate of  $20^\circ\text{C}/\text{min}$

Stages	Sample (EFB : POME sludge)	E (kJ/mol)	A, (1/s)	Kinetic Model	$R^2$
1	100 : 0	116465.31	$1.40 \times 10^{18}$	F3	0.9910
	90 : 10	96486.29	$7.21 \times 10^{14}$	F3	0.9968
	75 : 25	91946.74	$1.44 \times 10^{14}$	F3	0.9948

	50 : 50	80956.20	$1.02 \times 10^{12}$	F3	0.9882
	25 : 75	70726.41	$2.32 \times 10^{10}$	F3	0.9795
	0 : 100	66627.50	$1.51 \times 10^9$	F3	0.9467
2	100 : 0	89327.76	$2.77 \times 10^7$	F1	0.9986
		79405.60	$9.29 \times 10^5$	R3	0.9972
		168487.26	$2.41 \times 10^{13}$	D3	0.9976
	90 : 10	81203.96	$4.25 \times 10^6$	F1	0.9914
		194161.51	$7.63 \times 10^{15}$	D5	0.9972
	75 : 25	81272.14	$4.43 \times 10^6$	F1	0.9970
		193804.00	$7.50 \times 10^{15}$	D5	0.9971
	50 : 50	74111.12	$1.03 \times 10^6$	F1	0.9992
		65676.36	$4.65 \times 10^4$	R3	0.9961
		141008.83	$7.36 \times 10^{10}$	D3	0.9968
	25 : 75	68966.29	$3.25 \times 10^5$	F1	0.9990
		61002.95	$1.62 \times 10^4$	R3	0.9940
		131688.61	$9.77 \times 10^9$	D3	0.9952
	0 : 100	63473.91	$8.39 \times 10^4$	F1	0.9967
		154203.47	$1.53 \times 10^{12}$	D5	0.9987
3	100 : 0	167905.27	$1.55 \times 10^{12}$	F3	0.9836
		202126.52	$1.32 \times 10^{14}$	F3	0.9785
		176801.46	$2.64 \times 10^{12}$	F3	0.9753
		173550.61	$3.46 \times 10^{12}$	F3	0.9774
		169509.91	$1.25 \times 10^{12}$	F3	0.9971
	0 : 100	211313.71	$1.39 \times 10^{15}$	F3	0.9790

Stage 1 mainly involved the dewatering of samples, where the third order chemical reaction model (F3) was in dominion. The exceptions to this model are EFB : POME sludge of 25 : 75 and 0 : 100, which have higher correlation coefficients attributed to second order (F2) and one and a half order (F3/2), respectively. A lower rate of reaction for the two exceptions might be caused by the volatilizing of other small percentage components that was present in POME sludge. At this stage, the rate determining step was the chemical reaction, mostly dehydration reaction.

Hemicellulose and cellulose devolatilizes at Stage 2, which was the main decomposition stage of the samples. At this stage, many reactions were involved, such as devolatilization, cracking, hydration, and carbonization. Therefore, it was obvious that mass transfer had a dominant role in rate determination in addition to reaction. In Stage 2, the solid-state reaction followed first order kinetic (F1), and three dimensional diffusion mechanisms, either Jander equation (D3) or Zhuravlev, Lasokin, Tempelman equation (D5). Jander equation (D3) is for reactions in a sphere, where diffusion in all three directions is equally important [13]. On the other hand, Zhuravlev, Lesokin, Tempelman equation (D5) does not reveal the shape of the particle. Phase boundary mechanism or power law (R3) was dominant for EFB pyrolysis and in some co-pyrolysis ratios. Power law in phase boundary reaction (R3) is a reaction controlled by movement of an interface at constant velocity and at which nucleation occurs virtually immediately, so that the surface of each particle is covered with a layer of the product. This function is usually assumed to be the governing conversion model in the combustion of certain carbonaceous materials [13]. The rate determining mechanism for first order kinetic (F1) is the chemical reaction, where there was an equal probability of nucleation at each active site [13].

Both power law (R3) and Jander (D3) mechanisms involve reaction for spherical symmetries, suggesting the presence of spherical particles in the samples. Since both of these kinetic models were attributed to EFB but not POME sludge, it can be concluded that the sphere particles were contributed by EFB. Similar results were obtained by Liu *et al.*, in the study of the decomposition of wood and leaf of fir plants, which gave good linearity for first order reaction (F1), Jander equation (D3), and power law in phase boundary reaction (R3) [14].

For stage 3, devolatilization of lignin occurred for all samples and the model obtained was the third order kinetic model (F3). Employing Coats-Redfern method, Bu *et al.*, concluded that second order reaction mechanism (F2) fits well for raw alkali lignin pyrolysis with  $R^2$  of 0.9970. However, third order reaction mechanism also showed a good  $R^2$  of 0.9960 [15].

According to Yorulmaz and Atimtay, the fact that thermal analysis allows the fitting of more than one kinetic model to the samples is not favourable and was noticed in this study [16]. Thus, combining TGA including dynamic and isothermal studies could be used to obtain the exact mechanisms and thermal constants of the oxidation process. In the current study, third order (F3) mechanism was assumed to be the main mechanism responsible for the oxidation of samples in both Stage 1 and Stage 3. As for Stage 2, first order reaction (F1) is seen as the main mechanism, accompanied by power law (R3) and Jander equation (D3) or Zhuravlev, Lasokin, Tempelman equation (D5) mechanisms.

As observed from the Table 3, activation energies for the Stage 1 decreased with the increase in POME sludge percentages. The alkaline earth metals in sludge ash might have acted as catalyst and reduced the activation energy for volatilization. Besides that, the amount of volatiles decreases with the increase of POME sludge percentages, which in turn lowered the activation energy of the sample.

As the percentages of POME sludge increased in the sample, the temperature at which the main decomposition stage (Stage 2) started decomposing increased. This finding had been obtained by Mu *et al.*, [4]. It was explained that the percentage of volatiles in the samples played a role in contributing to the activation energy, where the activation energy increases with the increase in volatile percentage. As observed in Figure 1, both TG and DTG curves showed that the devolatilization stage of EFB is indeed much steeper than that of POME sludge, indicating the much higher volatile percentage in EFB.

As compared to other stages, Stage 3 that mainly involves the devolatilization of lignin showed the highest activation energy. The activation energy obtained was  $183.53 \pm 18.46$  kJ/mol. Decomposition of lignin required higher activation energy than other components as lignin has a more complicated structure and higher molecular weight. Besides that, char that formed in Stage 2 may have contributed to the higher activation energy due to secondary cracking.

## CONCLUSION

EFB showed higher reactivity regarding the higher mass loss rate as compared to that of POME sludge in thermogravimetric studies. Upon co-pyrolysis conducted, a positive synergistic effect (higher reactivity) was observed in the experiments than theoretical analysis. The mechanisms involved in the pyrolysis of EFB, POME sludge, and their blends were determined using

Coats-Redfern method. For Stage 1, the dominant kinetic model was the third order reaction (F3); for Stage 2, were first order reaction (F1), power law in phase boundary reaction (R3), and Jander equation (D3) or Zhuravlev, Lasokin, Tempelman equation (D5); for Stage 3, was third order reaction (F3). Activation energies for each stage were also analysed and it can be concluded that Stage 3 showed the highest activation energy, with an average value of  $183.53 \pm 18.46$  kJ/mol.

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## PROFILES



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## SYNERGIC EFFECT AND KINETIC MECHANISMS FOR CO-PYROLYSIS OF EMPTY FRUIT BUNCH AND PALM OIL SLUDGE



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