Thermogravimetric kinetic analysis of in-situ catalytic 
pyrolysis of palm oil wastes with the presence of palm oil 
wastes ash catalyst

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Abstract. The thermal degradation and kinetic analysis for oil palm frond (OPF) and oil palm trunk (OPT) with its ashes were investigated using thermogravimetric approach (TGA). OPF ash, OPT ash and its mixtures are used as a natural source of catalysts in the pyrolytic conversion of the palm oil wastes to bioenergy. The TGA experiments were conducted in a range of heating rates of 10-100 K/min from the temperature of 323 K to 1173 K. Coats-Redfern model is applied in this study to evaluate the activation energy ($E_a$) and pre-exponential factor ($A$). The average $E_a$ values ranged 24.27-32.36 kJ.mol$^{-1}$ and 41.42-46.10 kJ.mol$^{-1}$ for pyrolysis of OPF and OPT respectively. Meanwhile, the average $E_a$ values ranged 24.27-31.06 kJ.mol$^{-1}$ and 31.77-43.45 kJ.mol$^{-1}$ for catalytic pyrolysis of OPF and OPT respectively.

1. Introduction

Renewable energy is an alternative energy to reduce environmental impacts produced on this earth. From the various types of renewable energy resources, biomass energy is chosen in this present study.
as it is said to be easily available, does not rely with the climate change and the topography of the country as compared to wind, solar and hydroelectric energy [1]. Besides that, biomass does not released carbon dioxide (CO₂) to the environment as it will absorbs the CO₂ during the process for electricity generation. Furthermore, biomass is considered as the clean energy and heating sources. The production rate of biomass is high and can be easily be obtained to form as one of the energy resources compared to other renewable energy resources.

In this work, palm oil wastes such as oil palm frond (OPF) and oil palm trunk (OPT) were selected as the biomass for pyrolysis process due to its abundant availability in ASEAN countries particularly in Malaysia and Indonesia. It was reported an increasing trend of palm oil production in Indonesia these recent years and successfully achieved an estimated production of 34.5 million tons in year 2016 [2,3]. Meanwhile, an estimated of 19.16 million tons of palm oil production were recorded in Malaysia for that same year. As a result, around 43-45 % of biomass was generated from the palm oil plantation [4].

Palm oil biomass can potentially be used as a biofuel to replace fossil fuels production. It is reported that palm oil produced the highest yield oil when compared with other vegetable oils [5]. There are many industries built that utilise palm oil biomass as a feedstock in the aim of biofuel production and electricity generation [6]. In this work, OPF and OPT are selected as the oil palm biomass in the pyrolysis process. It is reported that the pre-exponential factor (A) of 1.2 x 10¹⁷ - 1.1 x 10¹⁷ min⁻¹ and activation energy (E_a) of 160 to 199 kJ.mol⁻¹ can be achieved in OPF and OPT in pyrolysis process [7, 8]. The presence of chemical composition such as hemicellulose, cellulose, and lignin content provides a potentiality as a source of fuel for thermochemical and biochemical conversion. The chemical composition for OPF in terms of hemicellulose, cellulose and lignin content are reported to be in 31.93 %, 42.12 % and 26.05 % respectively [9]. Meanwhile, the hemicellulose, cellulose, and lignin in OPT are 30.36 %, 50.78 % and 17.87 % respectively [9].

There are many studies found in literature using palm oil wastes in pyrolysis process with the presence of commercial catalyst. Cheah et al. [10] had investigated the catalytic pyrolysis for OPF with the presence of commercial HZSM-5 zeolite and graphite nanofiber (GNF). The authors had discovered that GNF catalyst performed better than HZSM-5 zeolite in the OPF pyrolysis by achieving a conversion performance between 66.57 wt% and 72.40 wt% [10]. Lim and Andrésen [11] had studied on the catalytic effect of boric oxide on empty fruit bunches (EFB) and OPF in a fixed pyrolysis bed reactor. It is reported that boric oxide has the ability to remove the hydroxyl and methoxy groups in the bio-oil with an efficiency of 50 % - 80 % [11]. Kabir et al. [12] conducted a comparative study of palm oil mesocarp fiber and OPF in a slow-heating fixed reactor (2.2 cm x 35 cm) with alkali metal as catalyst. It is reported that the highest bio-oil yield produced is 50 wt% and 47 wt% for palm oil mesocarp fiber and OPF, respectively at temperature 550 °C and 600 °C [12]. The role of the catalyst enables the reaction temperature to be lowered, quality of pyrolysis products can be improved in terms of stability and reduction of the oxygen compounds, and increased of product selectivity [13]. Few examples of different catalysts utilised in pyrolysis process are molecular sieve, metal oxides, transition metals had been evaluated on the bio-oil yields and properties [14].

To the authors best knowledge, it is discovered that the kinetic analysis for catalytic of OPF and OPT pyrolysis utilising its ashes as a natural catalyst had not been conducted before. However, there are studies conducted using rice hull ash in pyrolysis of rice husk [15] and coal bottom ash in pyrolysis of rice husk [16]. Therefore, in this work, the kinetic analysis for pyrolysis of OPF and OPT are investigated with the absence and presence of OPF ash, OPT ash, and OPF/OPT ash using thermogravimetric approach (TGA). The catalysts such as OPF ash, OPT ash, and OPF/OPT ash are utilised in the pyrolysis process of palm oil wastes to enhance the biomass to biofuel production. Coats-Redfern model (iso-conversional) is used to evaluate the kinetic parameters.

2. Experimental materials and methods

2.1. Biomass preparation
Both OPF and OPT are obtained from a local palm oil mill, Woodman, Miri Sarawak. Both of these biomass are dried using an oven until the moisture content are less than 10 wt% and grounded into powder form in a particle size less than 500 µm. The ultimate analysis for OPF are 40.5 wt% carbon, 6.8 wt% hydrogen, 3.5 wt% nitrogen, 0.3 wt% sulphur, and 48.9 wt% oxygen using LECO CHNS-932 elemental analyzer. The ultimate analysis for OPT are 42.4 wt% carbon, 7.3 wt% hydrogen, 0.3 wt% nitrogen, 0.7 wt% sulphur, and 49.3 wt% oxygen.

2.2. Catalyst preparation

The catalysts that are used for pyrolysis process are from the ashes produced from the combustion process of OPT and OPF through continuous heating process with temperature of 973 K for 4 hours using Laboratory Muffle Furnace, Carbolite ELF 11/14B. The ashes of OPT and OPF are grounded to fine particle sizes less than 500 µm. Thereafter, the ashes are then sent to oven for 12 hours at 373 K for drying purpose and left to cool down to 25 °C prior for TGA analysis.

2.3. Thermogravimetric analysis (TGA) approach

Pyrolysis experiments are conducted using a thermogravimetric analyser (TGA-DSC 1, Mettler Toledo). An approximate of 10 mg of samples are placed in the ceramic crucible under non-isothermal condition. The samples are pyrolysed from room temperature to 1173 K at different five heating rates of 10 K.min⁻¹, 20 K.min⁻¹, 30 K.min⁻¹, 50 K.min⁻¹, and 100 K.min⁻¹. Thereafter, the temperature of 1173 K is maintained for 10 minutes when it reached 1173 K. The biomass are mixed with the ashes in the weight ratio of 1:1 meanwhile the biomass/OPF ash/OPT ash samples are blended in the weight ratio of 1:0.05:0.05.

2.4. Kinetic theory

The kinetic principles that used in pyrolysis process are based on iso-conversional principle. This principle indicates the rate of the reaction will be affected by the temperature only with a basis of constant conversion rate [17]. Thus, thermal degradation of the lignocellulose component in biomass will occurred simultaneously within a conversion rate of the time, proposed a reaction mechanism of the pyrolysis process as shown as below equation (1).

\[
\text{Biomass (Palm oil waste)} \xrightarrow{k} \text{Volatile} + \text{Char}
\]

where \( k \) refers to pyrolysis conversion rate constant in transforming the biomass (palm oil waste) into volatiles and char. According to Sadhukhan et al. [18], the conversion rate of the pyrolysis process is assumed as single step process which follows Arrhenius and show in equation (2) as shown below.

\[
k = A e^{\left(\frac{-E_a}{RT}\right)}
\]

where \( A \) represents pre-exponential factor (s⁻¹), \( E_a \) represents activation energy (kJ.mol⁻¹), \( R \) represents the universal gas constant (8.314 J.(K.mol)⁻¹) and \( T \) represents the absolute temperature in Kelvin (K). The degradation rate of the reaction can be further expressed in equation (3) by showing the rate conversion of the biomass from solid to volatile state.

\[
\frac{d\alpha}{dt} = k(T) \cdot f(\alpha)
\]
The time rate of degradation or conversion, $\frac{d\alpha}{dt}$, represents the conversion rate which is temperature-dependant and $\alpha$ is the degree of conversion fraction of the sample in the respective time. The degree of conversion fraction is defined as the decomposition rate of the biomass sample at any range of the temperature in thermogravimetric analysis which is expressed in equation (4) as shown below.

$$\alpha = \frac{m_i - m_t}{m_i - m_f}$$  \hspace{1cm} (4)

where $m_i$ represents the initial mass of biomass sample, $m_t$ represents the mass of the sample at the given time, $t$, and $m_f$ represents the final mass of sample.

By substituting equation (2) into equation (3) to obtain equation (5) as depicts below.

$$\frac{d\alpha}{dt} = Ae^{\left(\frac{-E_A}{RT}\right)} \cdot f(\alpha)$$  \hspace{1cm} (5)

Non-isothermal method is commonly used in a solid-state kinetics where the heating rate, $\beta$ is remain constant and defined in equation (6).

$$\beta = \frac{dT}{dt}$$  \hspace{1cm} (6)

By combining both equations (5) and (6), which gives equation (7) as follows:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} e^{\left(\frac{-E_A}{RT}\right)} \cdot f(\alpha)$$  \hspace{1cm} (7)

By integrating equation (7) above with the function of $g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)}$, which simplifies the equation for numerical approximation solving and gives equation (8)

$$g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta} \int_{T_0}^{T} e^{\left(\frac{-E_A}{RT}\right)} dT$$  \hspace{1cm} (8)

In iso-conversional kinetics, the pyrolysis kinetic order is specified under first order reaction, which gives equation (9) as shown below.

$$g(\alpha) = -\ln (1 - \alpha)$$  \hspace{1cm} (9)

2.5. Kinetic models

Coats-Redfern model is the selected kinetic iso-conversional model to evaluate the kinetic parameters of the biomass pyrolysis in this study. The equation of the models will be linearised and plotted in a linear graph to verify the values of $A$ and the $E_A$ of the reaction. Table 1 shows the linear expression of
Coats-Redfern models with the respective x-axis, y-axis, y-intercept and slope. By plotting y-axis versus x-axis of each model, the $E_A$ can be obtained from the slope of the graph while the pre-exponential can be obtained from the y-intercept of the graph.

Table 1. Linear expression of three different models

| Model        | Equation                                                                 | y-axis          | x-axis          | Equation |
|--------------|--------------------------------------------------------------------------|-----------------|-----------------|----------|
| Coats-Redfern| $\ln\left(\frac{-\ln(1 - \alpha)}{T^2}\right) = \ln\left(\frac{AR}{\beta E_A}\right) - \frac{E_A}{RT}$ | $\ln\left(\frac{-\ln(1 - \alpha)}{T^2}\right)$          | $\frac{1}{T}$ |

3. Results and discussion

3.1. Thermal degradation behaviour of OPF and OPT in pyrolysis process

In the present study, the thermal degradation effect of the OPF and OPT with or without the presence of catalyst were analyzed by using thermogravimetric analysis (TGA) equipment. Figure 1 shows the curves of the thermogravimetric (TG) and derivative thermogravimetric (DTG) of OPF and OPT in non-catalytic and catalytic pyrolysis reaction with five different types of heating rates 10 to 100 K.min$^{-1}$. The TG curve is used to determine the weight loss of the sample in weight percentage with the increased of temperature meanwhile the DTG curve is used to analyse the initial, maximum and final biomass degradation temperature. Based on this figure, the trend of the curves can be categorized into three main stages of thermal degradation namely, Stage I: dehydration, Stage II: main devolatilisation and Stage III: decomposition of carbonaceous components.

Stage I of thermal degradation for OPF and OPT pyrolysis started from room temperature to 495 K and 500 K respectively with a slight reduction of weight fraction of OPF and OPT. This is where the dehydration process takes place to reduce the moisture content and light volatiles of the component [7]. The surface tension will tend to cause evaporation to both moisture content and the water bounded of the OPF and OPT biomass surface. When the temperature reached the end of Stage I decomposition, the internal forces such as intermolecular and intramolecular forces start to break the hydrogen bond in lignocellulose component and form water molecules to generate the char. When most of the moisture content in the OPF and OPT have been evaporated, the weight of the OPF and OPT decreases dramatically in all five different heating rates. This indicated that the biomass had experienced Stage II decomposition in temperature range from 495 to 750 K and 500 to 710 K for OPF and OPT respectively. This is the phase where the organic compound of OPF and OPT is undergoing main devolatilisation process for carbon and volatile component extraction. In this stage, transglucosidation process is happened where the intermediate cellulose component starts to degrade to levoglucosan and oligosaccharides [19]. In stage III, a slight weight loss of OPF and OPT with the temperature range from 750 to 1173 K and 500 to 1173 K respectively due to occurrence of passive devolatilisation process. Passive devolatilisation process is where the carbonaceous component started to decompose and both CO and CO$_2$ started to form by vaporisation of non-volatile carbon compounds that contained in the solid residue of the biomass [20]. Besides that, the aromatisation and bond cracking happens to convert the cellulose and carbonaceous material to char and volatile matter.

Figure 1 shows that there are two exothermic peaks in between the temperature range from 500 to 820 K for both OPF and OPT occurred in this curve for the five different heating rates. A lateral shift to the right hand side in the thermogravimetric curve was observed as the heating rates increases. The first peak occurred due to the thermal decomposition of protein and carbohydrates component while the decomposition of lignin happened in the second peak. The second peak for both OPF and OPT are relatively higher than the first peak due to high content of lignin content in oil palm wastes. It is where the main decomposition stage happened in the second decomposition stage as mentioned in the
description for the TG curve. In last stage (stage III), a small endothermic peak indicates that the remaining residue undergoes final decomposition stage after the temperature reached 820 K. Based on figure 1, the optimum mass loss was observed to be in the stage II where the temperature ranged from 495 to 750 K. The mass loss was observed in the average range of the residues weight (%) from the initial temperature, \( T_{\text{initial}} \) until the final temperature of stage II, \( T_{\text{final}} \) for the main pyrolysis stage (stage II).

3.2. Effect of heating rates on OPT and OPT thermal decomposition

It is said that high heating rate of the process will favors the depolymerisation of hemicellulose and cellulose and volatile cracking in secondary reactions. Additionally, the volatile compound in biomass will decreased due to the generation of polyhydroxy aromatics component [21]. Fast pyrolysis usually used high temperature for the process in order to generate high yield of bio-char, oil and non-condensable gases. From figure 1, it is seen that both TG and DTG curves increased with heating rates. The trend is in similar behavior with other biomass as reported by Soon et al. [7], Khan et. al [8], Fong et al. [22], Liew et al. [23], Loy et al. [24], Majid et al. [25] and Teng et al. [26].

3.3. Effect of catalyst on OPF and OPT thermal decomposition

This study enhances the important of catalytic pyrolysis process using renewable resources such as OPF ash and OPT ash catalyst. Chen et al. [20] reported that the EA of the palm oil pyrolysis was decreased significantly using rice husk ash as the natural catalyst in representing CaO catalyst. The main purpose of the study is to enhance the reusability of the oil palm wastes and improved the bio-fuel production [5].

In this present study, the EA of OPF was reduced from 28.49 kJ.mol\(^{-1}\) to 23.66 kJ.mol\(^{-1}\) with the addition OPF/OPT ash. Furthermore, the DTGmax reduced significantly from 5.74 to 4.43×10\(^{-2}\) %.min\(^{-1}\) at the heating rate of 100 K.min\(^{-1}\). A similar behavior is observed for OPT pyrolysis when comparing with literature findings by Liu et al. [21] and Chen et al. [27]. This can be further explained that the OPF/OPT ash is able to transform the complicated reaction into a semi simple-complex reaction involving less EA. The semi-complex reaction refers to the reagent involved in the system which can move freely meanwhile the activated complex have restricted movement.
Figure 1. TG and DTG graphs of (a) non-catalytic pyrolysis of OPF, (b) non-catalytic pyrolysis of OPT, (c) pyrolysis of OPF using OPF ash catalyst, (d) pyrolysis of OPT using OPT ash catalyst, (e) pyrolysis of OPF using OPF/OPT ash catalysts (f) pyrolysis of OPT using OPF/OPT ash catalysts.
3.4. Kinetic study

Coats-Redfern, an is conversional method was selected to identify the kinetic mechanism involved in the OPF and OPT pyrolysis with the presence of OPF ash and OPT ash as catalyst in this study. Based on the TG curve, the main devolatilization stage (Stage II) was selected as the main pyrolysis stage for both OPF and OPT in five different heating rates respectively. This model is applied to determine the kinetic parameters such as $E_A$ and $A$ for the pyrolysis of OPF and OPT in the absence and presence of catalyst. The role of $E_A$ determines the amount of energy that reactants need to overcome the pathway before the chemical reaction meanwhile $A$ represents the frequency of the collisions of the chemical reactants molecules during pyrolysis reaction [21]. The values of $E_A$ and $A$ were calculated based on the slope and intercept point of the kinetic plot. It was found that the average of $E_A$ and $A$ for pure OPF were 28.49 kJ mol$^{-1}$ and 1.12×10$^6$ min$^{-1}$. With the presence of OPF ash, the values of $E_A$ and $A$ are slightly reduced to 28.02 kJ mol$^{-1}$ and 1.15×10$^6$ min$^{-1}$. With the presence of OPF/OPT ash catalyst, the values of $E_A$ and $A$ obtained have been reduced to 23.66 kJ mol$^{-1}$ and 1.16×10$^6$ min$^{-1}$ respectively when compared without catalyst. On the other hand, the average of $E_A$ and $A$ for pure OPT were 44 kJ mol$^{-1}$ and 5.78×10$^6$ min$^{-1}$ respectively. With the presence of OPT and OPF/OPT ash, the values of $E_A$ and $A$ reduced to the range of 36.86 – 38.86 kJ mol$^{-1}$ and 1.14×10$^6$ - 1.16×10$^6$ min$^{-1}$. The presence of these biomass ash catalysts (OPF ash, OPT ash, and OPF/OPT ash) in the pyrolysis process enhances the rate of secondary reaction. The diffusivity from the OPT and OPF molecules will takes place on the porous structure of these biomass ash catalysts, followed by the metal oxides and char components existed in these biomass ash catalysts which allow the continuation of the degradation reaction. According to Xu et al. [20], they mentioned that good catalytic effect reduces the values of $E_A$ with increasing rate of reaction in the pyrolysis process.

Table 2 shows the average correlation coefficients ($R^2$) values for Coats-Redfern model for five different heating rates. It was observed that the $R^2$ values were above 95 % for the studied heating rates and conclude that this model is suitable for kinetic analysis with high accuracy of the results. As seen in table 2, the average values of the $E_A$ and $A$ for OPF pyrolysis are observed to be 28.49 kJ mol$^{-1}$ and 1.12×10$^6$ min$^{-1}$. Besides that, it was observed that the values of $E_A$ and $A$ increased with increasing heating rates from 10 to 100 K.min$^{-1}$ which is in good agreement with the findings found by Soon et al. [7]. It is found that the presence of OPF/OPT ash in OPF pyrolysis had reduced both the $E_A$ and $A$ values. The $E_A$ values had been reduced from 28.02 to 23.66 kJ mol$^{-1}$ and $A$ values had been reduced from 1.15×10$^6$ to 1.11×10$^6$ min$^{-1}$ when OPF/OPT ash is added in OPF pyrolysis. On the other hand, the average values of the $E_A$ and $A$ for OPT pyrolysis are observed to be 44 kJ mol$^{-1}$ and 5.78×10$^6$ min$^{-1}$ respectively. With the presence of OPF/OPT ash catalyst in the OPT pyrolysis, the values of $E_A$ and $A$ were increased from 36.86 to 38.86 kJ mol$^{-1}$ and 1.14×10$^6$-1.16×10$^6$ min$^{-1}$ respectively. The observation of the results found in this study is in accordance with the literature as reported by Gan et al. [28] where the $A$ were range from 1.1×10$^6$ min$^{-1}$ to 189.4×10$^6$ min$^{-1}$ using Coats-Redfern model. It was observed that the pyrolysis of OPF with OPF/OPT ash catalyst gave the lowest $E_A$ and $A$ for all samples using Coats-Redfern model.
Table 2. Kinetic evaluation for the catalytic and non-catalytic pyrolysis of OPF and OPT using Coats-Redfern model.

|          | $\beta$ (K.min$^{-1}$) | $E_A$ (KJ.mol$^{-1}$) | $A$ (min$^{-1}$) | $R^2$   |
|----------|-------------------------|------------------------|------------------|---------|
| OPF      | 10                      | 24.27                  | 2.43x10$^5$      | 0.9881  |
|          | 20                      | 26.79                  | 5.09x10$^5$      | 0.9955  |
|          | 30                      | 28.81                  | 7.85x10$^5$      | 0.9976  |
|          | 50                      | 30.22                  | 1.34x10$^6$      | 0.9980  |
|          | 100                     | 32.36                  | 2.74x10$^6$      | 0.9961  |
| Average  |                         | 28.49                  | 1.12x10$^6$      | 0.9951  |
| OPF -    | 10                      | 24.27                  | 2.24x10$^5$      | 0.9950  |
| OPF ash  | 20                      | 28.14                  | 5.26x10$^5$      | 0.9936  |
|          | 30                      | 26.77                  | 7.84x10$^5$      | 0.9901  |
|          | 50                      | 31.06                  | 1.38x10$^6$      | 0.9920  |
|          | 100                     | 29.89                  | 2.82x10$^6$      | 0.9792  |
| Average  |                         | 28.02                  | 1.15x10$^6$      | 0.9700  |
| OPF -    | 10                      | 18.66                  | 2.19x10$^5$      | 0.9672  |
| OPF/OPT  | 20                      | 22.13                  | 4.87x10$^5$      | 0.9871  |
| ash      | 30                      | 26.05                  | 7.88x10$^5$      | 0.9911  |
|          | 50                      | 24.44                  | 1.29x10$^6$      | 0.9906  |
|          | 100                     | 27.02                  | 2.75x10$^6$      | 0.9887  |
| Average  |                         | 23.66                  | 1.11x10$^6$      | 0.9849  |
| OPT      | 10                      | 41.42                  | 2.44x10$^5$      | 0.9878  |
|          | 20                      | 46.10                  | 4.57x10$^5$      | 0.9871  |
|          | 30                      | 43.97                  | 7.48x10$^5$      | 0.9762  |
|          | 50                      | 44.48                  | 1.26x10$^6$      | 0.9636  |
|          | 100                     | 44.00                  | 2.67x10$^6$      | 0.9504  |
| Average  |                         | 44.00                  | 5.78x10$^6$      | 0.9730  |
| OPT -    | 10                      | 38.89                  | 2.68x10$^5$      | 0.9795  |
| OPT ash  | 20                      | 34.60                  | 5.42x10$^5$      | 0.9715  |
|          | 30                      | 33.61                  | 8.64x10$^5$      | 0.9612  |
|          | 50                      | 43.45                  | 1.29x10$^6$      | 0.9614  |
|          | 100                     | 33.73                  | 2.87x10$^6$      | 0.9160  |
| Average  |                         | 36.86                  | 1.16x10$^6$      | 0.9579  |
| OPT -    | 10                      | 31.77                  | 2.72x10$^5$      | 0.9430  |
| OPT/OPF  | 20                      | 39.71                  | 5.34x10$^5$      | 0.9664  |
| ash      | 30                      | 40.77                  | 7.89x10$^5$      | 0.9976  |
|          | 50                      | 40.13                  | 1.37x10$^6$      | 0.9590  |
|          | 100                     | 41.92                  | 2.76x10$^6$      | 0.9417  |
| Average  |                         | 38.86                  | 1.14x10$^6$      | 0.9615  |

4. Conclusions
A kinetic analysis for pyrolysis of various palm oil wastes such as oil palm frond (OPF), and oil palm trunk (OPT) was successfully conducted with the absence and presence of OPF ash, OPT ash, and OPF/OPT ash via thermogravimetric approach. Coats-Redfern model had successfully achieved the regression coefficients above 95 % in pyrolysis process utilizing these feedstock. The degradation results
were found in this sequence: OPF-OPF/OPT ash > OPF-OPF ash > OPF > OPT-OPT ash > OPT-OPT/OPT ash > OPT.

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