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Catalytic Pyrolysis of Torrefied Empty Fruit Bunch (EFB) with Sulphated Zirconia

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ABSTRACT

In this study, empty fruit bunch (EFB) was utilised as a feedstock in the pyrolysis process using a fixed-bed reactor in the presence of a sulphated zirconia catalyst after it underwent a torrefaction process in a furnace. The optimisation of process parameters such as catalyst to biomass ratio (factor A) and nitrogen flow rate (factor B) was investigated through Fractional Factorial Design (FFD) using Stat-Ease Design Expert software version 7 with bio-oil yield considered as the response. Eleven experimental runs were performed at a fixed pyrolysis temperature of 500°C and a 20 K/min heating rate. Analysis of Variance (ANOVA) analysed the results. The model was statistically significant. Factor A and the interaction between AB were significant parameters with a p-value < 0.05. The value of R^2 was 0.8951, which indicated that the selected factors and their levels showed a good correlation to the bio-oil yield from catalytic pyrolysis of the torrefied EFB process. A linear model equation was developed to predict the highest bio-oil yield. The highest bio-oil yield of 42.07 wt% was achieved when the reaction was performed without the catalyst and at a nitrogen flow rate of 100 cm^3/min.

Keywords: *Empty fruit bunch, torrefaction, catalytic pyrolysis, optimisation, fractional factorial design.*

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INTRODUCTION

Biomass is a potential renewable energy since it has low emissions of pollutants and carbon neutrality [1]. Malaysia is blessed with the plantation of an oil palm tree to produce crude palm oil, which is significant to the Malaysian economy. During crude palm oil production, biomass waste such as empty fruit bunch (EFB), palm frond and mesocarp fibre are co-generated. Approximately 15.8 -17.0 million tonnes of empty fruit bunch (EFB) was generated over the years [2]. Since EFB are renewable and abundantly available throughout the year, it has the potential as a source of energy.

Biomass pyrolysis is a thermochemical process at a temperature range of 400- 600°C without an oxidising agent, which transforms biomass to bio-oil, combustible gas and solid char [3]. Pyrolysis is an attractive method to convert biomass to high-value-added products [4]. Bio-oil from biomass pyrolysis has the potential to be utilised as a fuel. However, bio-oil has high oxygen content (40–50 wt%), high water contents (15–30 wt.%) and low H/C ratios of bio-oil that restrict its direct utilisation as transportation fuels directly [5, 6]. This indicated that the raw biomass has high moisture and oxygen content. Therefore, it has to undergo certain pretreatment steps, such as torrefaction, to convert the biomass into torrefied biomass with a lower amount of oxygen and water contents [7, 8]. Torrefaction is conducted in an inert atmosphere at a temperature range of 200-300°C that targets the moisture and volatiles elimination from a hemicellulosic fraction of biomass [9].

Catalytic pyrolysis is also one method to improve bio-oil qualitatively and quantitatively through oxygen removal and its stability [5]. Catalytic pyrolysis is an approach to upgrade the quality of bio-oil from non-catalytic pyrolysis also been reported [5, 10, 11]. The process usually employs a zeolite-type catalyst because it can assist in deoxygenation reaction during biomass pyrolysis. Catalytic pyrolysis of biomass with zeolites type catalyst has been reported [12-15]. However, there are several drawbacks to incorporating zeolite in catalytic pyrolysis of biomass processes, such as being prone to coking [16]. Therefore, there is a need to find an alternative catalyst, such as a metal oxide type with similar properties to zeolite.

Several studies have reported the use of metal oxide catalysts in biomass pyrolysis. It was recorded that $Ca(OH)_2$ and MgO catalysts had increased the phenolic content and reduced the undesirable acidic components in bio-oil from catalytic pyrolysis of raw EFB [17]. The presence of Na_2CO_3, $ZnCl_2$ and Al_2O_3 in *Anchusa azurea* pyrolysis improved the liquid yield [10]. However, the presence of $Ca(OH)_2$. Na_2CO_3 has resulted in a lower liquid yield. CaO, MgO and ZnO were incorporated in the fast pyrolysis of cellulose at 500 ^{o}C, and it was reported that the CaO catalyst had exhibited the best performance in reducing the acidity of bio-oil [18].

Sulphated zirconia is a highly acidic catalyst that exhibits catalytic activity and catalyses many organic reactions due to its strong acidity [19]. Their acidity is much higher than that of

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concentrated H_2SO_4, with their surface sulfate groups acting as Lewis and Brønsted sites with thermal stability [20]. It was reported that sulfated zirconia (SO_4^{2-}/ZrO_2) favoured the formation of furan in catalytic pyrolysis of cellulose using pyrolyser (py-GCMS) [19]. In another study, it was reported that the solid acid sulfated zirconia (SO_4^{2-}/ZrO_2) efficiently promoted the pyrolysis of cellulose to produce levoglucosenone at low temperatures [21]. At a higher temperature of approximately 500 ^oC, sulphated zirconium hydroxide was reported to be active in the pyrolysis of polypropylene, low-density polyethene and high-density polyethene for the increasing liquid yields [22].

The literature regarding the catalytic pyrolysis of raw biomass with metal oxide is substantial. However, knowledge of the catalytic pyrolysis of torrefied biomass, especially EFB with sulphated zirconia, must be improved. The catalytic pyrolysis of torrefied EFB with sulphated zirconia has to be studied to determine whether the catalyst significantly affects bio-oil yield. In this paper, torrefaction of EFB was performed in a furnace under a limited oxygen environment, followed by pyrolysis and catalytic pyrolysis with sulphated zirconia in a fixed-bed reactor.

EXPERIMENTAL

Biomass pretreatment

The EFB was obtained from North Star Palm Oil Mills Kuala Ketil, Kedah. Tap water was used to rinse the biomass for impurities removal purposes. Then, the EFB was dried in an oven at 80°C for 24 hours. Afterwards, the sample was shredded and sieved using a Retsch sieve shaker. The sieved EFB in the range of 500-710 µm was selected for the torrefaction process.

Proximate analysis of EFB

The proximate analysis of the EFB was conducted according to the American Standard Testing Materials (ASTM) with particle size in the range of 125-250 µm. The moisture content was determined according to ASTM E871-82, while volatile matter and ash content were determined following ASTM E872-82 and ASTM D1102-84, respectively. Fixed carbon content was estimated by difference.

Torrefaction of EFB

Approximately 10.0 g of EFB was placed into a furnace, and the temperature was set at 280°C for 30 min for the torrefaction process. The reaction temperature was set at 280°C since most

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hemicellulosic fractions underwent decomposition [8]. After the experiment, the crucible was cooled at room temperature. Finally, the sample was collected and weighed.

Preparation of catalyst

Sulphated zirconia has been prepared using a one-step sol–gel method using zirconium propoxide. About 5 ml of $Zr(OCH_2CH_2CH_3)_4$ solution (Aldrich, 70 wt% in 1-propanol) mixed with 6.6 ml of 1-propanol (Aldrich, 99.5%). Aqueous sulfuric acid (0.5 M) solution has been prepared separately, and 9.7 ml of the aqueous sulfuric acid solution was added dropwise to the solution of $Zr(OCH_2CH_2CH_3)_4$ in 1-propanol with vigorous stirring for at least 6 hours; a gel formed. The gel was filtered, dried at 100°C, and calcined at 625°C for 4 hours [23].

Pyrolysis and catalytic pyrolysis in a fixed-bed reactor

Catalytic pyrolysis of torrefied EFB with sulfated zirconia was performed in a fixed-bed reactor. In common pyrolysis, approximately 5.0 g of sample was placed in the reactor tube. The amount of catalyst loading and nitrogen (N_2) flow rate were set according to the suggested conditions by DOE software. Initially, 100 cm^3/min inert N_2 was purged into the reactor for 30 mins to provide an inert environment and function as sweeping gas, rapidly removing hot gases from the reaction zone into the condensation apparatus. The catalytic pyrolysis was operated at a pyrolysis temperature of 500°C for 30 min with 20K/min of heating rate. Bio-oil was quenched in the condenser and collected in the receiver flask, immersed in a mixture of ice and water for each experiment. The related equation to calculate bio-oil yield is shown in (1).

$$\text{Bio-oil yield (wt\%)} = \frac{\text{mass of bio}-\text{oil (g)}}{\text{mass of EFB (g)}} \times 100\% \qquad (\mathbf{1})$$

Design of Experiment

A 2 level-factorial-design, 2^2, with a catalyst to biomass ratio, A (0-20 wt.%) and nitrogen flow rate, B (100-500 cm^3/min), were employed. These two factors were used to correlate with the response (R1), which was the bio-oil yield (%). Twenty-two with replicated twice, and three centre points corresponded to 11 experimental runs were suggested.

RESULTS AND DISCUSSION

The proximate analysis of EFB has been conducted with particle size in the range of 125-250µm. The comparison between the current study and the reported literature is shown in Table 1. In this study, sample EFB contains 8.29 % moisture content which is in good agreement with Nyakuma et al. [24] and Abdullah et al. [25]. The volatile matter content is 72.04 % which is slightly lowered compared to 82.53 and 83.86 %, as reported by Nyakuma et al. [24] and Abdullah et al. [25], respectively. The ash and fixed carbon contents of EFB are 5.5 % and 14.17 %, which are in the range reported by Abdullah et al. [25].

Table 1: The proximate analysis of EFB

Moisture Content (%)	Volatile Matter (%)	Ash Content (%)	Fixed Carbon Content (%)	Reference
8.29	72.04	5.50	14.17	This study
8.43	82.53	4.97	4.08	[22]
7.95	83.86	5.36	10.78	[23]

DOE analysis

The results for bio-oil yield from pyrolysis and catalytic pyrolysis of torrefied EFB with sulphated zirconia using a 2-level factorial design, 2^2, are tabulated in Table 2.

Table 2: The experimental results for bio-oil yield

Run	Catalyst to biomass ratio (wt.%)	Nitrogen flow rate (cm^3/min)	Bio-oil yield (wt%)
1	0.0	100.0	42.89
2	0.0	100.0	41.22
3	0.0	500.0	36.95
4	0.0	500.0	38.77
5	10.0	300.0	37.37
6	10.0	300.0	36.38
7	10.0	300.0	36.93
8	20.0	100.0	33.47
9	20.0	100.0	35.39
10	20.0	500.0	36.96
11	20.0	500.0	34.80

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The analysis of variance (ANOVA) is tabulated in Table 3. The model p-value of 0.0024 indicated that the model is statistically significant. The values of p-values used to check for the significance of each of the parameters studied. The values of p-values < 0.05 indicated that the model terms are significant. In this case, A and AB are significant model terms with p-values of 0.0010 and 0.0125, respectively. The curvature p-value of 0.4211 implied that the curvature in the design space is not significant. Therefore, a straight line that represents the model is suggested. There is a 42.11% chance that a curvature F-value this large could occur due to noise. The model equation for the bio-oil yield in terms of coded factors is as follows:

$$\text{Bio-oil yield} = 37.56 - 2.40*A - 0.69*B + 1.41*A*B \qquad (2)$$

Table 3: Analysis of Variance (ANOVA) for the FFD model

Source	**Sum of Squares**	**Degree of Freedom**	**Mean Square**	**F Value**	**p-Value**	**Remarks**
Model	65.83	3	21.94	17.06	0.0024	Significant
A -Catalyst to biomass ratio	46.13	1	46.13	35.86	0.0010	Significant
B – Nitrogen flow rate	3.77	1	3.77	2.93	0.1379	Not significant
AB	15.93	1	15.93	12.39	0.0125	Significant
Curvature	0.96	1	0.96	0.75	0.4211	Not significant
Pure Error	7.72	6	1.29	-	-	-

R^2 value = 0.8951

The experimental R^2 value is 0.8951. This indicates that non-dependable parameters show a good correlation with dependable parameters. In this case, the selected parameters, the catalyst to biomass and nitrogen flow rate with its selected range, have a good contribution to the response, which is the bio-oil yield. The straight line represents the regression for the error from replicates (Figure 1). Factor A and AB are essential since their p-values are 0.0010 and 0.0125, respectively. Both factors are shown far from the regression line for the error from replicates.

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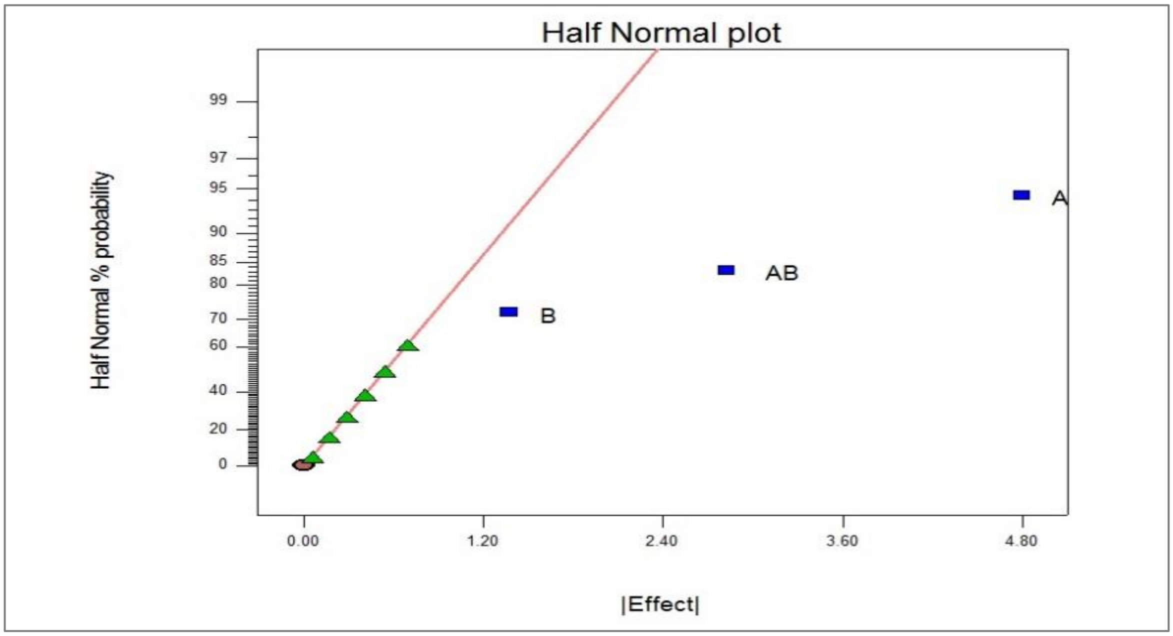

Figure 1: The half-normal plot

Interaction between catalyst-to-biomass ratio and nitrogen flow rate

Figure 2 shows the interaction between factor A and factor B towards bio-oil yield. Figure 2 shows the interaction between parameters A (catalyst to biomass ratio) and B (nitrogen flow rate). A maximum bio-oil yield was attained at a low catalyst-to-biomass ratio (0 wt) and low nitrogen flow rate (100 cm3/min). At point A, the bio-oil yield is the highest (42.89 wt%) compared to all points B, C and D, where the bio-oil yields were 37.87, 35.88 and 34.43 wt%, respectively. Point A is when no catalyst is involved (0 wt %), and the nitrogen flow rate is 100 cm^3/min. Point B shows the bio-oil yield at the same catalyst-to-biomass ratio but a higher nitrogen flow rate of 500 cm^3/min.

The difference between bio-oil yields at points A and B could happen due to insufficient condensation of the hot vapour by the cooling apparatus in the system. It is identified that the higher flow rate had caused the rapid removal of products from the reaction medium [26, 27]. Both of these reasons might contribute to lower bio-oil yield. It is observed that bio-oil yield at point A is higher, which indicates that at lower nitrogen flow rates, the velocity of sweeping gas is slower to transfer the hot vapour into the condensation section, which gives sufficient time for condensable gases to be transferred and condensation process to occur.

It is determined that the pyrolysis of torrefied EFB produced a higher amount of bio-oil than the reaction involving the catalyst. Sulphated zirconia catalyst has reduced the condensable product yield due to thermal cracking of molecules, but producing more non-condensable gas and coke. This finding is similar to [18], which reported the utilisation of catalysts such as CaO, MgO and ZnO that do not affect the cellulose degradation for bio-oil formation.

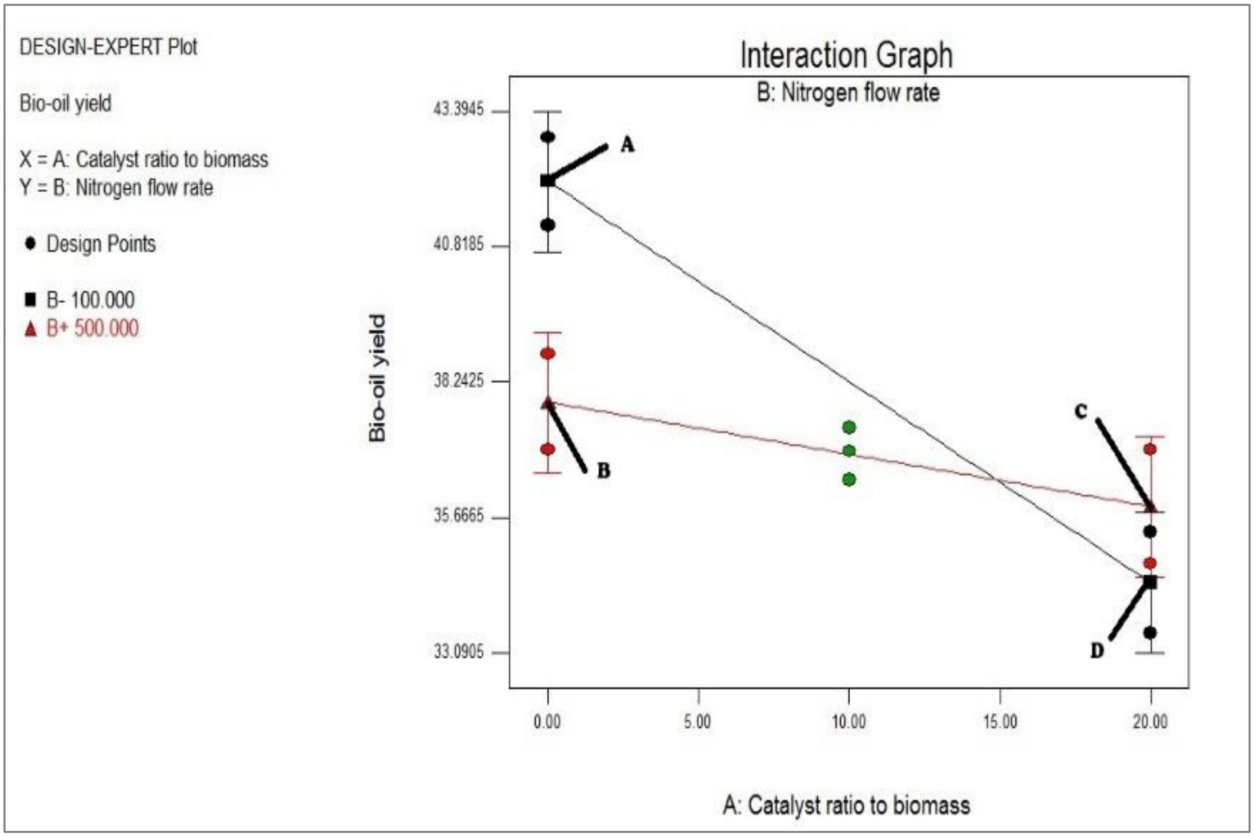

Figure 2: The effect of interaction between factor A and factor B towards bio-oil yield. Points C and D are the catalyst ratio at 20wt% at different nitrogen flowrate of 500 and 100 cm^3/min, respectively.

Optimal design

The optimal design is carried out to optimise the conditions of bio-oil yield obtained from the torrefied EFB pyrolysis. The parameters involved, such as catalyst to biomass ratio and nitrogen flow rate, are set in the range, maximise, minimise, target or equal to provide several solutions of a suitable combination of factors to obtain the highest bio-oil yield. The set value for each of the variables is tabulated in Table 4.

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Table 4: The optimal design for pyrolysis of torrefied EFB

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Catalyst ratio to biomass	Minimise	0	20	1	1	3
Nitrogen flow rate	Minimise	100	500	1	1	3
Bio-oil yield	Maximise	33.47	42.89	1	1	3

Test runs were carried out at the optimised condition of catalyst to biomass ratio of 0 wt% and nitrogen flowrate of 100 cm^3/min to obtain a theoretical bio-oil yield of 42.05 %, with the desirability of 0.970. The desirability of 0.970 is accepted because it is the only suggested optimised condition by the software. Five replicates were performed at these conditions. The predicted and observed values are shown in Table 5. The average experimental bio-oil yield is 42.07 %. The experimental and predicted values are almost similar. From the calculated standard deviation, the value of 1.557 indicates the high degree of agreement between the experimental values.

Table 5: The bio-oil yield from optimisation conditions

Run	Catalyst to biomass ratio	Nitrogen flow rate (cm^3/min)	Response-Bio oil yield (%)	
			Observed	Predicted
1	0.00	100.0	43.82	42.05
2	0.00	100.0	42.08	42.05
3	0.00	100.0	39.60	42.05
4	0.00	100.0	42.04	42.05
5	0.00	100.0	42.80	42.05

CONCLUSION

Pyrolysis and catalytic pyrolysis of torrefied EFB with sulphated zirconia catalyst in a fixed bed reactor has been performed. FFD was employed as a tool to investigate the parameters that play significant roles in the pyrolysis process. Catalyst to biomass ratio and interaction between catalyst-to-biomass ratio was discovered as significant factors through ANOVA analysis. The maximum bio-oil, 42.07 %, was obtained when the reaction was performed without a sulphated

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zirconia catalyst and a nitrogen flow rate of 100 cm^3/min. Catalytic pyrolysis of torrefied EFB has produced a higher amount of coke and non-condensable gases instead of bio-oil. This can be concluded that the sulphated zirconia catalyst has reduced the condensable product yield due to the thermal cracking of molecules.

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AUTHOR’S CONTRIBUTION

Alina Rahayu Mohamed and Nur Nasulhah Kasim conceptualised the central research idea, provided the theoretical framework, and supervised the research progress; Nur Annashuha Zakry performed the experimental part of the research and wrote the article. Nur Nasulhah Kasim and Razi Ahmad contributed to the editing and reviewing process and revisions.

CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted without any self-benefits or commercial or financial conflicts and declare the absence of conflicting interests with the funders.

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