



Investigation of palm fibre pyrolysis over acidic catalyst for bio-fuel production

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Abstract

This research aimed to evaluate the pyrolysis reaction for bio-fuel production from palm fibre. A preliminary step to investigate the effect of different type of heterogeneous catalyst revealed three different catalysts: zeolite type (H-Beta), tungsten–zirconia (WO_3/ZrO_2) and modified alumina ($\text{KOH}/\text{Al}_2\text{O}_3$). An increase in temperature was found to have a positive impact on the production of bio-fuel. Bio-fuel synthesis performed in a stainless steel tubular reactor was found to optimized at 650 °C using WO_3/ZrO_2 as a catalyst resulting in 40.5% bio-fuel, 7.1% bio-char and 14.3% gas. Formation of gas product is due to the presence of strong acid sites on the surface of WO_3/ZrO_2 promoting cracking reactions. H-Beta catalyst produced the largest amount of bio-char which is also due to strong acid sites and narrow catalytic pore structure which caused rapid deposit of coke and then formation of bio-char. Pyrolysis of palm fibre over $\text{KOH}/\text{Al}_2\text{O}_3$ catalyst produced the least amount of bio-fuel, but contain the largest composition of phenolic compounds. These compounds are the transformed from lignin content in palm fibre on alkaline sites of the $\text{KOH}/\text{Al}_2\text{O}_3$ catalyst. A larger scale production facility was designed to produce larger amount of bio-fuel for the engine performance test. The biofuel blended gasoline (10% biofuel) was tested in an eight-cylinder spark-ignition engine. Engine performance testing revealed that the brake power and torque generated from combustion of bio-fuel blended oil was lower than conventional gasoline. The brake specific fuel consumption of bio-fuel blended was slightly higher than conventional gasoline.

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1. Introduction

Two main problems that our communities have encountered daily is the accumulation of organic waste and energy crisis due to shortage in petroleum reservoir. These two problems can be simultaneously solving by converting waste

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Nomenclature

B_{fuel}	Synthesized bio-fuel (%)
C_{solid}	Synthesized char material (%)
G_c	Gas generated (%)
R_{fibre}	Remaining palm fibre after reaction (%)

into a renewable source of energy. Pyrolysis process thermally decomposed raw material into bio-char, bio-fuel, and synthetic gas in the absence of oxygen [1]. Additionally, lignin content inside lignocellulosic biomass can be transformed into phenol molecule [2]. These intermediates can then be delivered to electricity generating facilities to provide a clean source of energy. The type of output from a pyrolysis process depends mainly on the kind of raw material used as a feedstock and the pyrolysis operating condition. For instance, pyrolysis of animal fat at 500 °C (heating rate of 5 °C per min) produced mostly bio-char and bio-oil [3]. Chen et al. reported an increase in selectivity toward liquid product when palm's fibre used as feedstock was compressed into a pellet before being fed into a pyrolysis fixed-bed reactor. Additionally, a pyrolysis process when operated under CO₂ environment instead of N₂ generated a larger amount of liquid product [4]. Combination of corn cob and waste cooking oil as feedstock for co-pyrolysis process was found to generate 24.96 MJ/kg of bio-char, 32.78 MJ/kg of bio-oil, and 16.06 MJ/Nm³ of synthetic gas [5].

Pyrolysis of palm fibre was also investigated in batch and fixed bed reactor. Hameed et al. prepared zeolite from palm fibre which was then used in the pyrolysis reaction converting palm fibre into bio-oil at 550 °C [6]. HZM-5, which is a type of zeolite catalyst, was employed for pyrolysis of rice straw [7]. Optimization of bio-oil production from palm fibre using horizontal fixed-bed reactor was found at 700 °C [8]. Dias et al. demonstrated kinetic modelling of the pyrolysis of wood material. It was revealed that activation energy of the reaction increases with lignin content. It was also documented in their research that pyrolysis reaction is confined to mass/heat transfer limitation instead of kinetic control restriction [9]. Mass transfer constraint is an important obstacle to the pyrolysis process that required the use of catalyst. This could be problematic because most pyrolysis required catalyst to accelerate the decomposition reaction.

Heterogeneous catalyst is used mainly in reaction inside high pressure and high temperature fixed-bed reactor. For instance, Chiaramonti et al. reported the use of catalyst for the pyrolysis of waste cooking oil to bio-oil. Catalytic pyrolysis at 500 °C and 4 h⁻¹ weight hourly space velocity resulted in as high as 64 wt% bio-oil product [10]. Other catalysts included HY zeolite which was used in the co-pyrolysis of corn cob and waste cooking oil. An increase in the acid concentration of HY zeolite was found to increase the production of bio-oil [11]. The type of acid site also influences the pyrolysis reaction. Incorporation of precious metal such as tungsten on the acidic catalyst was found to increase the concentration of Lewis acid site, which enhanced the pyrolysis process [12,13]. However, one of the main limitations of heterogeneous catalysts is their stability [14,15]. For this reason, it is important for researchers to increase the pore size of the acidic catalyst in order for the pyrolysis process to be properly improved. Pinard et al. developed a desilication method which was proven to significantly increase the pore size of HZM-5 zeolite [16]. Another modification technique used to create a hierarchical porous catalyst was dealumination, which has been performed on H-Beta zeolite [17] and HZM-5 zeolite [18] by hydrogen fluoride solution.

The aim of this research was to investigate the influence of operating conditions such as reaction temperature, type of catalyst and pressure on the composition of pyrolysis products consisting of bio-char, bio-oil and synthetic gas. The pyrolysis reaction was conducted in a fixed-bed reactor using palm fibre as feedstock and different types of heterogeneous catalyst (H-beta, tungstate-zirconia, KOH doped Al₂O₃). Characterization of heterogeneous catalysts included N₂ sorption and NH₃ temperature desorption. Bio-oil characterization included FTIR and acidity analysis by using ASTM D664 technique. Derived bio-char was analysed using N₂-adsorption, thermogravimetric analyser and a bomb calorimeter.

2. Method and apparatus

2.1. Pyrolysis reaction

Palm fibres from empty fruit bunches were obtained in bulk from Palm Oil Factory in Surathani province, Thailand. Before used palm fibres were washed with 1 M of sodium hydroxide in warm water and then dried

in an oven at 110 °C overnight. Approximately 10 g of palm fibres were ground (300 mesh), mixed with catalyst and then loaded into a vertical fixed-bed reactor on quartz wool. The reactor was filled with ceramic balls up to the top. Helium gas (9.99%) as carrier gas was delivered into the reactor system at 70 mL/min measured by a built-in digital flow metre. Bio-oil was collected at the bottom of the reactor using glass condenser attached to a water circulation cooler.

2.2. Bio-oil analysis

Composition both in terms of quality and quantity of the bio-oil product was analysed using GS-MS device (Agilent 5977B) equipped with a HP-INNOWAX column (30 m length \times 300 μ m in diameter). The device was attached to a computer, which contained a software used to display data, analysed results of relative similarity compared with databased, control the operating parameters. Through this software the injection temperature was set at 170 °C, split ratio of 100:1 and helium flow rate of 200 mL/min. The temperature programme started at 45 °C for 10 min and then ramped up at the rate of 5 °C/min to 290 °C and held isothermally for 20 min. The percentage composition of the main component phenol and benzene derivatives were obtained by integration of the area under the peaks, which was calibrated by known composition of both components. (See Fig. 1.)



Fig. 1. GS-MS device (Agilent 5977B) used for analysing biofuel.

The amount of bio-fuel (B_{fuel}), bio-char (C_{solid}), gas (G_{c}) and fibre remaining (R_{fibre}) can be calculated from the equations below.

$$B_{\text{fuel}} = \frac{\text{Amount of biofuel collected}}{\text{Amount of feedstock}} \times 100 \quad (1)$$

$$C_{\text{solid}} = \frac{\text{Amount of biochar collected}}{\text{Amount of feedstock}} \times 100 \quad (2)$$

$$G_{\text{c}} = 100 - B_{\text{fuel}} - C_{\text{solid}} - \left(\frac{\text{Amount of biofuel collected}}{\text{Amount of feedstock}} \times 100 \right) \quad (3)$$

2.3. Engine performance testing

This part of the research was performed to evaluate the outcome of the bio-oil produced based on actual testing inside an eight-cylinder spark-ignition engine. The engine belonged to an old model of Ford automobile. Specification of the testing engine are shown in Table 1. An aluminium compartment is used to cover the engine to shield the engine from external environment.

Bio-oil synthesized from the pyrolysis reaction was blended with gasoline (benzene) along with 1% emulsion agent. The code name for the blended bio-oil is B10Z90 which represented 10 wt% bio-oil and 90% gasoline. A dynamic dynamometer using AC electric was used to simulate load for the engine. A computer was used to control

Table 1. Engine specification using for testing bio-oil.

Engine characteristics	Value
Number of cylinders	8
Displacement volume	0.607 L
Type of engine	Naturally aspirated and direct injection
Bore × stroke	101.6 mm × 76.2 mm
Manifold type	Dual plan carb
Manufacture brake power	164.1 kW @ 4000 rpm

Table 2. Composition of products from pyrolysis of palm shell fibre using different type of catalyst.

Catalyst °C	B _{fuel} (%wt)			C _{solid} (%wt)			G _c (%wt)			R _{fibre} (%wt)		
	450	550	650	450	550	650	450	550	650	450	550	650
No catalyst	16.8	22.4	25.4	1.2	1.4	1.2	3.7	2	0.9	78.3	74.2	72.5
H-Beta	25.7	34.3	23.4	9.4	12.3	13.8	11.6	8.2	22.5	53.3	45.2	40.3
WO ₃ /ZrO ₂	22.4	39.5	40.2	6.2	7.3	7.1	22	12.9	14.3	49.4	40.3	38.4
KOH/Al ₂ O ₃	9.3	22.7	24.1	2.3	3.2	2.3	8.1	0.7	1.4	80.3	73.4	72.2

the dynamometer, the workbench and temperature at various location (lubricant and coolant). An AVL IndiModul 622 equipment was employed to record the in-cylinder pressure and the flow of fuel was measured using AVL 753. The direct injection pump was used to deliver gasoline fuel inside the chamber at pressure of 100 bar. The AVL models are group of instruments which are widely used for sensing, recording and display data correlated with the performance of the engine. The specific details of the AVLS sensing equipment are as followed: sampling rate per channel of 14 bit/800 kHz per channel, resolution of 1 °CA, on board memory 64 MB, and analog Input Signal +/- 10 V. The logic procedures for this experiment is shown in Fig. 2.

3. Result and discussions

Pyrolysis of palm oil was performed inside a fixed bed reactor with different type of catalyst including zeolite type (H-Beta), tungsten–zirconia (WO₃/ZrO₂), and KOH/Al₂O₃. In the first set of experiment, temperature was varied from 350 °C to 650 °C. The production of bio-oil at different temperature and type of catalyst is shown in Fig. 3. An increase in pyrolysis temperature was found to significantly increase the amount of bio-oil produced. The results correlated well with other researches [7,19]. Pyrolysis over H-beta and WO₃/ZrO₂ were found to give the highest amount of bio-oil compared with KOH/Al₂O₃ and the control experiment with no catalyst. This is due to the presence of both bronsted and lewis acid sites on the catalyst surface which promote decomposition of cellulose, hemicellulose and lignin into bio-oil [20,21]. For H-Beta, the amount of bio-oil reached the peak at 550 °C and decreased from 34.2% to 26.3% when reaction temperature increased to 650 °C. This is due to the large amount of hard coke formation inside micropores of H-Beta causing the acid site inside the catalyst to be inaccessible [22]. Table 1 demonstrated the formation of biofuel, char and gas product. A 8.9% increase in bio-fuel amount in the product was observed when H-Beta catalyst was used compared with no catalyst reaction. Production of bio-fuel from H-Beta Large amount of char was found to be form over H-Beta, which is due to the narrow catalytic pores of the zeolite type catalyst causing isomerization and char formation [23]. Large amount of gas was form at higher temperature due to the presence of acidic sites. For H-Beta catalyst gas amount increased from 8% to as high as 22% as the pyrolysis temperature increased from 500 °C to 550 °C. A 17.8% increase in the bio-fuel amount was found after the pyrolysis temperature was increased from 450 °C to 650 °C over WO₃/ZrO₂ catalyst. The dramatic increase in bio-fuel amount when H-Beta was used was due to the performance of acidic site on the catalyst. (See Table 2.)

Fig. 4 demonstrated a reduction in benzene content inside the synthetic bio-oil. This is because benzene can be easily cracked into small and more volatile molecule on acid sites inside the zeolite and tungsten–zirconia catalyst [24,25]. KOH/Al₂O₃ resulted in the smallest amount of benzene at pyrolysis temperature in the range of 350 °C to 550 °C. The largest production of benzene from pyrolysis reaction was observed in the absence of catalyst. Fig. 5 illustrated the effect of temperature and type of catalyst on the formation of phenol via the pyrolysis of palm

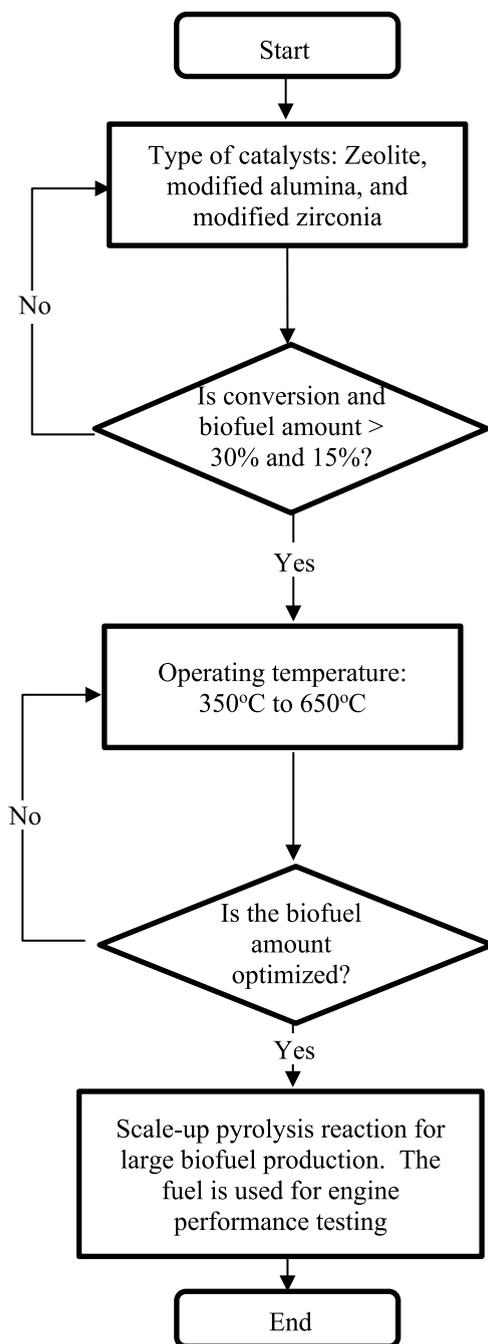


Fig. 2. Flow chart of the experimental process from type of catalysts, operating conditions, and engine performance testing.

fibre. Phenol component can be formed from the depolymerization of lignin content inside palm fibre [26]. Similar to other researches utilization of alkali catalyst was found to promote production of phenolic components [27–29]. An increase in pyrolysis temperature was found to slightly reduce the composition of phenolic compounds inside the bio-oil. This is because under the presence of alkaline catalyst, phenolic compounds can be converted to cresol compound at higher temperature due to deoxygenation reaction.

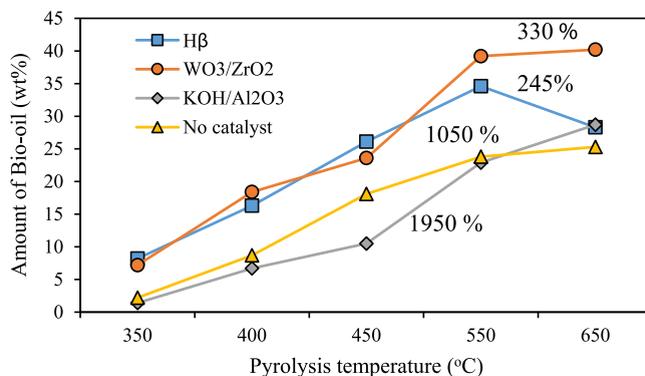


Fig. 3. Influence of pyrolysis temperature and type of catalyst on the amount of bio-oil produced from Palm fibre (operating conditions: helium atmosphere, 0.5 g catalyst and 2 h reaction time).

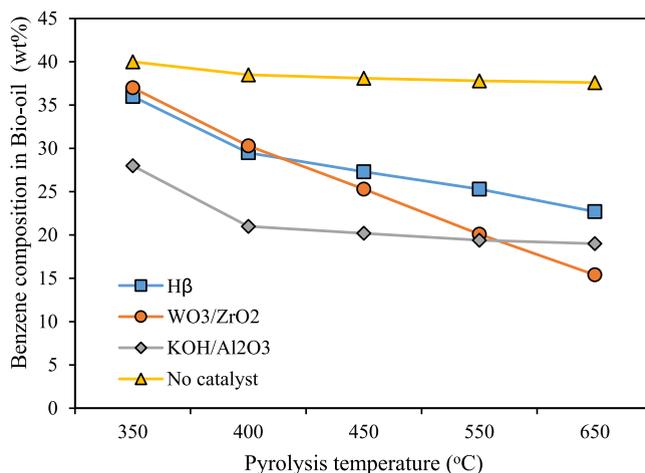


Fig. 4. Influence of pyrolysis temperature and type of catalyst on benzene composition in bio-oil produced under helium atmosphere and 0.5 g catalyst.

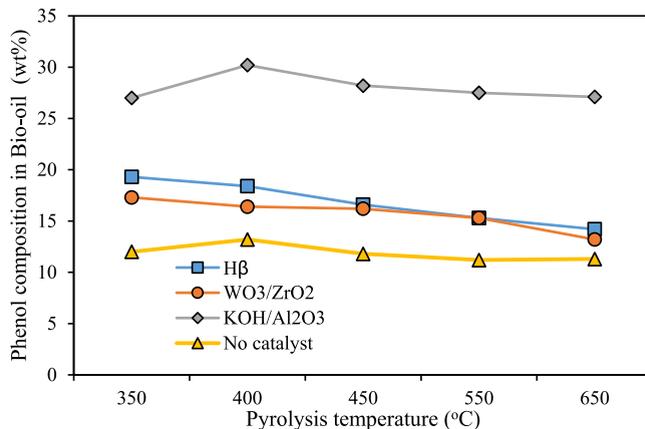


Fig. 5. Influence of pyrolysis temperature and type of catalyst on phenol composition in bio-oil produced under helium atmosphere and 0.5 g catalyst.

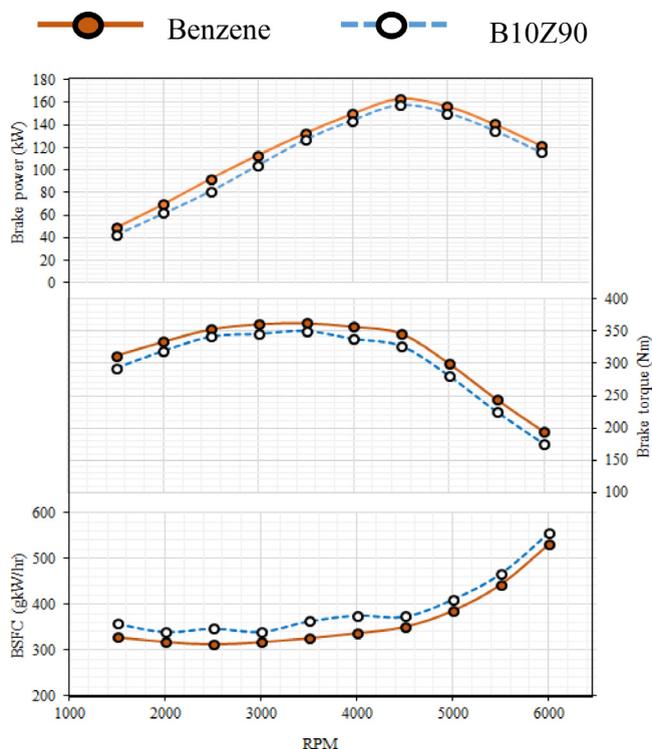


Fig. 6. Engine performance of the bio-oil (10 %wt) blended with benzene (90 %wt) compared with gasoline.

The optimum condition using WO_3/ZrO_2 catalyst was performed in order to produce large portion of bio-oil from palm fibre. At least 10L of bio-oil have to be used to test in the internal combustion engine workbench. Fig. 6 illustrated the result of engine performance testing based on torque, brake power and brake specific fuel consumption of the bio-oil (10%) blended with benzene (90%) fuel. The engine performance testing results was compared with pure benzene fuel. Brake power achieved from bio-oil blend peaked at 4500 rpm at 145 kW. Torque generated from bio-oil blend at 4500 rpm was 350 Nm and the brake fuel specific consumption of bio-oil at 4500 rpm was 376 g kW/h. Bio-oil blend demonstrated slightly lower brake power and torque compared with pure benzene fuel. Additionally, brake fuel specific consumption of bio-oil was slightly higher than pure benzene. The higher brake fuel specific consumption of bio-oil is because bio-oil have lower heating value compared with pure benzene fuel [30,31].

4. Conclusion

The objective of this research is to investigate the pyrolysis reaction over different type of heterogeneous catalyst and reaction temperature. Preliminary experimental revealed three practical catalysts for pyrolysis reaction including tungsten–zirconia (WO_3/ZrO_2), zeolite (H-beta) and modified alumina ($\text{KOH}-\text{Al}_2\text{O}_3$). The reaction temperature was varied from 350 °C to 650 °C. Production of bio-fuel was optimized at 650 °C over WO_3/ZrO_2 generating 40.5% bio-fuel, 7.1% bop-char and 14.3% gas. Pyrolysis over $\text{KOH}/\text{Al}_2\text{O}_3$ was the least effective in the production of bio-fuel. However, high composition of phenolic compound was observed in bio-fuel solution when the pyrolysis reaction was conducted over $\text{KOH}-\text{Al}_2\text{O}_3$ catalyst. This is due to the degradation of lignin content inside the palm fibre which transform into phenolic compound. The reaction system was scale-up to produced more bio-fuel at optimized condition. The produced bio-fuel was blended with gasoline solution (10%) and used in a spark–ignitions engine performance testing workbench attached to a dynamometer. Engine testing results demonstrated that the fuel blended with 10% bio-fuel have slightly lower brake power and torque compared with gasoline. The brake specific fuel consumption of the blended bio-fuel is slightly higher than conventional gasoline.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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