



Characterization of Bio-oil produced by green synthesized Zeolite- Y catalyzed pyrolysis of *Milicia excelsa*

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ABSTRACT

Pyrolysis-based bio-refinery technology have a huge potential to convert biomass residues into useful biofuels and chemical products. Liquid biofuels produced from biomass are becoming more popular due to the adverse environmental impacts of over-dependence on fossil fuels and its depletion. In this study, bio-oil was produced by green-synthesized zeolite –Y (GZY) catalyzed pyrolysis of *Milicia excelsa* (Me) residues at various temperatures (400, 550 and 700 °C) and biomass/catalyst (b/c) ratios (100/0, 80/20 and 60/40). Me residue was characterized by ultimate analysis while catalyst and bio-oil compositions were examined by Fourier transform infrared (FTIR), Gas Chromatography-Mass Spectrometry (GCMS) and X-ray fluorescence spectroscopy (XRF). Results of characterized bio-oil revealed that phenols, catechols and ketones were the significant compounds present. The highest percentage of phenols 59.59% was obtained at 550°C and b/c ratio of 60/40 with light aromatic compounds. The bio-oil's higher heating value is between 25.78 to 33.04 MJ/kg. These values are comparable with those of fossil diesel. Hence, this shows that Me fuel wood can be used substitute fuel for internal combustion engines in transportation.

1. INTRODUCTION

Energy security and sustainable development have been the major concerns that need urgent attention. Hence, scholarly studies are focusing on biomass utilization to produce fuels and chemicals to mitigate the effects of fossils while meeting the ever increasing energy demand (IEA, 2025; REN21, 2025; IRENA, 2026). In order to preserve our environment for future generations, the use of alternative fuels

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must be encouraged (Wang et al., 2017; Amhamed et al., 2024; Alli et al., 2024). One of the most available renewable resources is biomass, which provides a clean, renewable energy source that has the potential to greatly enhance the economy, environment, and energy security (Shendrik et al., 2021; Osman, et al., 2021; Ozor et al., 2022; Jegawa et al., 2025). When compared to fossil fuels, biomass offers significant social, political, and economic benefits. The transportation sector, internal combustion engines, and power plants all use biofuels (bio-char, bio-oil, and bio-gas. Utilizing biomass in combination with the right technology make it possible to produce hydrocarbons as a renewable energy source while also protecting the environment from issues related to the disposal of biomass wastes (Ozor et al., 2022; Fatonah et al., 2025). Conversion technique via pyrolysis for large-scale production of sustainable fuels (bio-char, bio-oil, and bio-gas) has become promising process and use of biomass. Phenolic, oxygenated organic, aromatic, and trace of sulfur and nitrogen-containing substances make up the complex mixture (bio-oil) obtained. Thermally breaking down of biomass at intermediate reaction temperatures (400–600 °C) in an inert atmosphere provide higher yield of bio-oil (60–70 wt%) (Oyebanji, et al., 2020; Osman, et al., 2021; Maneechakr & Karnjanakom, 2021; Oyebanji, et al., 2023). Drawbacks of using bio-oil include its high viscosity, low heating value, high corrosiveness, and thermal instability. It is necessary to upgrade the oxygenated pyrolysis vapor used as pyrolysis oil in internal combustion engines in order to reduce wear, corrosion, and pollution. Bed of catalysts can be used to pyrolyze the biomass once it has been combined with the feedstocks (Chukwuneke et al., 2019; Chuxia et al., 2019; Chanchangi et al., 2023; Mohan et al., 2025). Research on how zeolite-based catalysts shows that high temperatures promote the production of aromatic hydrocarbons (Aduba & Shimada 2023). Additionally, it has been observed that quality hydrocarbons are improved by upgrading oxygenated pyrolysis vapours with Zeolite-Y (Ezealigo et al., 2021a; Davidson, 2023). Because it is readily available as a raw material, non-corrosive, inexpensive, and produces high-quality selective hydrocarbons, the green zeolite-Y catalyst was employed in this investigation (Wang et al., 2017; Oyebanji, et al., 2020; Ezealigo et al., 2021b; Davidson., 2023; Oyebanji, et al., 2023). This study sought to determine how the pyrolysis reaction temperatures and biomass-to-catalyst ratios affect yields and quality of bio-oil from biomass residue (*Melicia excelsa* residue), and the authors are aware that no studies have been done on the use of green zeolite catalysts made from *Ficus exasperata* (sand paper) leaves for the catalytic pyrolysis of *Melicia excelsa* (Me) (IEA 2025; IRENA 2026).

2. MATERIALS AND METHODS

2.1 Materials

Me samples were obtained from local a wood processing facility in Ota, Nigeria, and green zeolite-Y was obtained. The material was pulverized and sieved into particles size 0.0 to 0.5 mm (0-500 µm) for further processing (Oyebanji et al., 2020).

2.2 Preparation of zeolite catalyst

The synthesis of green zeolite-Y from *Ficus exasperata* leaves (sandpaper leaves) include gel formation, aging and crystallization. The procured *Ficus exasperata* leaves was washed thoroughly and then sun dried. The dried leaves were calcined at 650 °C for 4 h to ash (ASTM D1102- 84). *Ficus Exasperata* Ash (FEA) (2.500 g, Merck) was added to polypropylene (PP) (2.000 g, Merck), sodium aluminate (NaAlO₂) (5.285 g, Sigma-Aldrich) and sodium hydroxide (NaOH) (0.294 g, Merck) in distilled water (76.620 g). The liquid was agitated magnetically for 45 minutes at 100 °C using a polyethylene rod before crystallizing for 45 minutes at 90 °C. After that, a stainless-steel autoclave lined with Teflon received the combined solution. After being mixed, the solution crystallized at 90–120 °C for 3–9 hours with the autoclave container closed. The samples were then removed, filtered, and rinsed with deionized

water until the pH of the filtrate reached 6-7 after the autoclave had naturally cooled to room temperature. Before further testing and characterizing the filtrate, it was dried using a muffle furnace overnight at 105 °C (ASTM C 566 - 19). The resulting nano particles was mixed with sawdust samples at a specified ratio to biomass.

2.3 Experimental Setup

The pyrolysis reactor comprised a 4 kW heater, gas collector, condenser, ice bath, Proportional-Integral-Derivative (PID) controller, and gas flow system. Experiments was carried-out in a fixed-bed reactor under an inert nitrogen atmosphere across a range of temperatures. The pyrolysis procedure was performed at predetermined furnace temperatures of 400, 550, and 700 °C, with nitrogen gas flowing at 100 mL min⁻¹ to purge the system of air, at specified heating rates (30 mins) and residence times of 5s. Biomass/catalyst feed ratios were determined by Box-Behnken experimental design generated using Minitab 17. Three feed configurations were evaluated: a non-catalytic run using 100 g of biomass alone, and two catalytic runs with biomass-to-catalyst ratios of 80:20 and 60:40, each tested across the designated temperature range.

2.4 Characterization procedures

2.4.1 CHN analysis

CHN analyzer (Perkin Elmer, 2400 Series-II) was utilized to determine carbon (C), hydrogen (H), nitrogen (N), and oxygen (O) in accordance with ASTM C566-19 and ASTM D1102 and Higher Heating Value (HHV) was estimated according to equation (1) (Sarkar & Wang, 2020):

$$HHV_{DRY} \left(\frac{Mj}{kg} \right) = -1.3675 + 0.3137C + 0.7009H + 0.0318O \quad (1)$$

Where: C, O and H, represent the percentage (dry basis) of carbon, oxygen and hydrogen, respectively.

2.4.2 GC-MS analysis

Extracted bio-oil was examined to GC-MS analysis using a Gas chromatograph analyzer model QP2010 at scanning range of 40 - 700 m/z⁻¹ in a helium atmosphere.

2.4.3 Determination of bio-oil properties

The flash point, density, pour point, viscosity, cloud point and pH values of bio-oil were determined. Flash point was determined using Tanaka Scientific Flash Point Tester with a thermometer attached to the power source. The tester was then placed into the cup containing the bio-oil sample as indicated by the filling mark. Using an external ignition source (lighter), the test flame was lit, and the coarse adjustment screw was used to adjust the flame. The process was repeated three times, and the bio-oil samples average flash point values were recorded (ASTM-D92). The bio-oil sample was partially filled into a 50 ml beaker, and the probe of a Controls digital thermometer (Model D1227) was submerged in the bio-oil to determine the cloud point. A 500 ml beaker filled with ice chunks was placed within a 50 ml beaker. When the bio-oil began to turn hazy and lost its transparency, the thermometer was closely monitored. The cloud point of the bio-oil was identified as the temperature at which cloudiness first appears. Their average readings were recorded after the process was done thrice (ASTM-D2500). After determining the cloud point, the pour point was ascertained by immersing the beaker containing the bio-oil further into the ice block. Every five seconds, the bio-oil's flow was monitored, and the moment at which the congealed bio-oil stopped flowing and solidified into an immovable solid was closely watched

and recorded (ASTM-D97). pH meter, Thomas Ostwald oil viscometer and density meter was used to measure acidity, viscosity and density at 35 °C laboratory room temperature.

3. RESULTS AND DISCUSSION

3.1 Elemental and proximate analysis of *Milicia excelsa* and *Ficus exasperata* (L.) leaf

Table 1 and Table 2 show the chemical composition of *Me* sawdust and catalyst bio-material [*Ficus exasperata* (L.)] leaves sample used in the experiment. Table 1 shows the elemental composition and proximate analysis of the selected wood sample. The percentages of nitrogen (0.35%) and sulfur (0.01%) show that the biomass sample is environmentally friendly and a suitable source for production of biofuels. The Sulphur content in *Me* is lower than the range reported in the literature for coal (0.5 - 5%) (Lin et al., 2015; Li, 2022; Oyeboji et al., 2023). Table 2 presents the elemental composition and proximate analysis of raw *Ficus exasperata* (L.) leaves.

Table 1. Elemental analyses of *Me*

Analysis	Element	Composition (%)
Ultimate Analysis (%)	C	30.96
	H	5.90
	N	0.35
	S	0.01
	O	62.78
Proximate Analysis (%)	Moisture Content	8.56
	Volatile matter	74.8
	Fixed Carbon	14.86
	Ash	1.78
Heating value	HHV (MJ/kg)	14.48
Molar Ratio	H/C	2.28
	O/C	1.52
	(O + N)/C	1.53

Table 2. Elemental and proximate analyses of *Ficus exasperata*

Ultimate Analysis		Proximate Analysis	
Elements	Composition (%)	Analysis	Composition (%)
C	48.50	Moisture Content	9.00
H	6.70	Volatile matter	74.45
N	0.20	Fixed Carbon	14.60
O	44.59	Ash	1.95
S	0.01		

3.2 Characterization of synthesized catalyst

XRF and FTIR analyses were used to examine the green zeolite -Y produced from *Ficus exasperata* leaves. Table 3 shows the sample's mineralogical characteristics and chemical make-up. The findings from XRF analysis agreed with the work of Oyeboji et al. (2020). This study found that calcinated *Ficus exasperata* leaves contained 67.5% silica and 6.5% alumina with SiO₂/Al₂O₃. The results are in

line with a number of previous studies that found SiO₂ (87.40%) and Al₂O₃ (3.60%) (Niu, et al., 2019); SiO₂ (67.10%–76.80%) and Al₂O₃ (3.43%– 5.69%) (Manadee *et al.*, 2017); SiO₂ (78.34%) and Al₂O₃ (8.55%); and SiO₂ (70.5%) and Al₂O₃ (5.23%) (Manadee *et al.*, 2017) (Shendrik et al., 2021). According to Gil-González *et al.* (2018), zeolitic minerals can be found and recognized using the FTIR spectrum between the asymmetric 1250 cm⁻¹ and 950 cm⁻¹ (Figure 1), while Si-O or Al-O bonds is responsible for frequencies around 1000 cm⁻¹, high crystalline zeolite and OH type functional group vibrations are seen in the highest bands of 3430 and 1608 cm⁻¹. Weak intensity wideband was seen at a wavelength of 565.41 cm⁻¹, indicating the presence of double-ringed zeolite crystallisation and the Y cubic prism band.

Table 3. XRF analysis of *Ficus exasperata* nano particules

Major Element (%)	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃	MnO
Sample ash	0.12	3.15	6.50	67.50	3.70	4.45	5.60	0.50	5.70	0.06
Average	0.09	1.2	2.2	25.5	0.5	0.5	27.0	<LD	6.0	00.2
Std.dev	0.01	0.01	0.02	0.58	0.01	0.01	0.02	<LD	0.02	0.01
Exp.value	2.03	4.08	8.73	25.4	1.02	2.22	26.04	0.02	13.3	1.02
SiO ₂ /Al ₂ O ₃ ratio	10:1									
Trace Element (Ppm)	Ba	S	Cl	Cu	Pb	Ni	Cr	Ce	Rb	Zr
Sample ash	440	1.2	0.2	30	22	20	15	42	55	45
Average	55.0	1.0	2.2	0.5	0.2	0.1	12.0	2.5	6.3	4.0
Std.dev	0.78	0.01	0.01	0.02	0.01	0.01	0.02	0.02	0.02	0.01
Exp.value	450.1	1.70	0.03	33.3	10.1	34.2	99.02	55.0	96.1	68.0

(Source: Author's compilation)

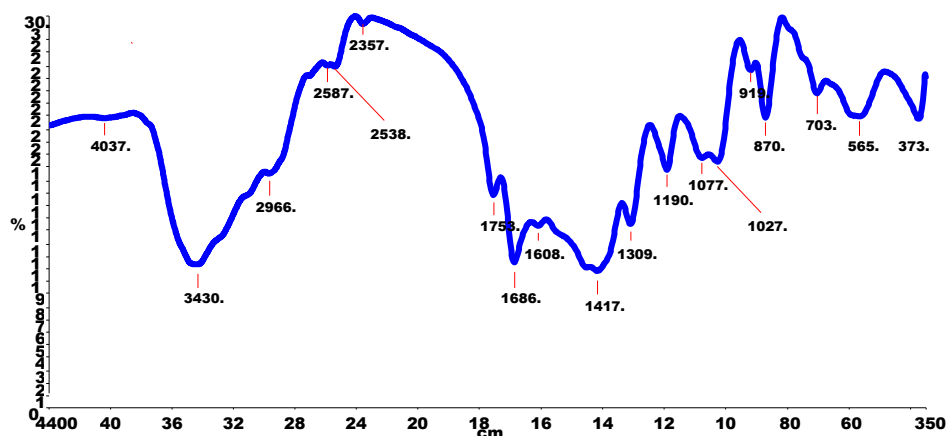


Fig 1. FTIR spectra of synthesized zeolite Y

3.3 GC-MS analysis of non-catalytic and catalytic Bio-oil

GC-MS analysis results of both non-catalytic and catalytic pyrolysis bio-oil are presented in Tables 4 – 7. The analysis results of non-catalytic pyrolysis bio-oil are shown in Table 4 with the presence of phenols, creosol, furans, ketone, carboxylic acid, esters and aldehydes, catechol and others (44.45, 4.85, 8.08, 15.6, 1.26, 3.52, 16.51 and 0.05%, respectively) at 400 °C, (45.73, 6.85, 4.91, 18.76, 4.36, 4.02, 9.94 and 0.05%, respectively) at 550 °C and (45.88, 5.85, 5.13, 9.29, 6.01, 3.23, 14.88 and 2.24%, respectively) at 700 °C. The phenol yields increased modestly but consistently with temperature, from

44.45 % at 400 °C to 45.73 % at 550 °C and 45.88 % at 700 °C. The gradual increase is due to progressive thermal degradation of lignin, and more lignin macromolecules are cleaved at ether and carbon-carbon bonds, releasing more phenolic fragments (Antony & Farid, 2022; Trubetskaya et al., 2023). Table 5 presents catalytic pyrolysis results at 400 °C, at b/c ratio of 80/20 as 46.59, 0.00, 0.00, 8.98, 4.34, 12.93, 13.43 and 10.84%, and at b/c ratio 60/40 as 51.43, 0.00, 0.00, 9.08, 5.07, 6.38, 15.65 and 11.40%. Phenol yields were increased from 44.45% (non-catalytic) to 46.59% (80/20) and 51.43% (60/40). The increase with increasing catalyst loading suggests that the catalyst was involved in the cracking and deoxygenation of oxygenated intermediates to stable phenolic structures.

Table 4. *Me* GC-MS identified non-catalytic compounds

Compound (%)	Biomass/ catalyst (100/0 g)		
	400 °C	550 °C	700 °C
Phenols	44.45	45.73	45.88
Creosol	4.85	6.85	5.85
Furans	8.08	4.91	5.13
Ketone	15.6	18.76	9.29
Carboxylic Acid	1.26	4.36	6.01
Esters and Aldehydes	3.52	4.02	3.23
Catechol	16.51	9.94	14.88
Others	0.05	0.05	2.24
Total	94.32	94.62	92.51

(Source: Author's compilation)

Table 5. *Me* GC-MS identified compounds at 400 °C

Compound (%)	Biomass/ catalyst (g)	
	80/20	60/40
Phenols	46.59	51.43
Creosol	0.00	0.00
Furans	0.00	0.00
Ketone	8.98	9.08
Carboxylic Acid	4.34	5.07
Esters and Aldehydes	12.93	6.38
Catechol	13.43	15.65
Others	10.84	11.40
Total	97.11	99.04

(Source: Author's compilation)

Table 6 shows the results of catalytic pyrolysis bio-oil at 550 °C for b/c ratio of 80/20 as 59.04, 0.00, 0.00, 13.57, 2.26, 4.72, 13.2 and 5.69%, and at b/c ratio 60/40 as 59.59, 0.05, 0.05, 12.68, 4.68, 7.70, 9.42 and 5.21%. The highest phenol yields under all conditions were obtained at 550 °C, at 59.04% (80/20) and 59.59% (60/40). catalyst convert furans, ketones and aldehydes into phenolic compounds through dehydration, decarbonylation and aromatization reactions. Table 7 shows the results of catalytic pyrolysis bio-oil at 700 °C for b/c ratio of 80/20 as 47.82, 0.00, 0.00, 12.40, 5.30, 7.48, 6.42 and 14.63% and at b/c ratio 60/40 as 48.07, 0.00, 0.00, 12.24, 5.20, 10.47, 6.50 and 12.68%, respectively. Phenol yields decreased slightly to 47.82% (80/20) and 48.07% (60/40) but were still higher than the non-catalytic levels. This decrease indicates that at higher temperatures the secondary cracking reactions

become dominant, breaking down some of the phenolic compounds into lighter gaseous products or polymerizing them into char and heavier residues.

Table 6. *Me* GC-MS identified compounds at 550 °C

Compound (%)	Biomass/ catalyst (g)	
	80/20	60/40
Phenols	59.04	59.59
Creosol	0	0.05
Furans	0	0.05
Ketone	13.57	12.68
Carboxylic Acid	2.26	4.68
Esters and Aldehydes	4.72	7.70
Catechol	13.2	9.42
Others	5.69	5.21
Total	98.48	99.38

(Source: Author's compilation)

Table 7. *Me* GC-MS identified compounds at 700 °C

Compound (%)	Biomass/ catalyst (g)	
	80/20	60/40
Phenols	47.82	48.07
Creosol	0.00	0.00
Furans	0.00	0.00
Ketone	12.40	12.24
Carboxylic Acid	5.30	5.20
Esters and Aldehydes	7.48	10.47
Catechol	6.42	6.50
Others	14.63	12.68
Total	94.05	98.16

(Source: Author's compilation)

The results also revealed that the presence of green zeolite-Y enhanced the process of aromatisation and favored hydrocarbons cracking to high amount of alumina and silica (Aghaei et al., 2020; Mishra et al., 2020; Sanchez et al., 2021; Al-Shafei et al., 2022). Optimal phenolic compounds (59.59%) yield was recorded for *Me* bio-oil at 550 °C with b/c ratio 60/40. The percentage composition of phenol yield in this study is higher than what was reported by Chroho *et al.* (2022), which was 7.9% phenols and 27.2% organic acids. Results in this study are consistent with those of several other researchers (Chukwuneke *et al.*, 2019; Sarkar & Wang, 2020; Ibrahim, et al., 2022; Chroho *et al.*, 2022; Oyekale, & John, 2023; Ungureanu et al., 2025). The compounds identified can be used to produce fuel, industrial chemicals and adhesives. Table 8 presents the basic thermo-physical properties, pour point, flash point, pH value, density, viscosity, and cloud point, of non-catalytic bio-oil at the optimal temperature (550 °C) as 72 °C, -12 °C, 6.4, 846 kgm⁻², 4.6 mm²/s, and -5 °C. This study found that the catalytic bio-oil acidity decreases with flash point and with increased viscosity very close to that of diesel at 80/20 biomass/catalyst (b/c) ratio with temperature 550 °C (Foroutan et al., 2019; Boichenko et al., 2025).

Table 8. Bio-oil Fuel properties at optimum Temperature (550 °C)

Bio-oil source	Properties	Diesel (Foroutan et al., 2019)	Heavy fuel oil	Biomass/catalyst (g)		
				100/0	80/20	60/40
<i>Milicia excelsa</i>	Flash point (°C)	75	-	72	79	86
	Pour point (°C)	-2	-	-12	-2	7
	pH	5.5-8	-	6.4	7.5	7.6
	Density (kgm ⁻³) 40°C	780	940	846	867	880
	Viscosity (mm ² /s) 40°C	1.8 -4.1	180	4.6	3.5	3.0
	Cloud point (°C)	30	40	-5	7	15

(Source: Author's compilation)

3.4 Physicochemical and biochemical properties of bio-oil yield

The physicochemical and biochemical composition of catalytic and non-catalytic bio-oil at the optimum temperature of 550 °C is shown in Table 9. The non-catalytic pyrolysis of *Me* produced very low hydrocarbon yields, the catalytic bio-oil yields showed general trends of increasing aromatic hydrocarbon yields, increasing phenol yields, and slightly decreasing yields of other compounds relative to the non-catalytic bio-oil. Increase in catalyst will reduce coke production (evidenced in lignin reduction) while significantly affecting other bio-oil characteristics. Carbon and fixed carbon of bio-oil increased as the catalyst increased with slight increase in sulphur and ash (Chukwuneke *et al.*, 2019; Okekunle *et al.*, 2021; Ungureanu *et al.*, 2025). Low ash concentration of bio-oil improves thermal and combustion efficiency in ICE applications while reducing the risk of corrosion, erosion, and incrustation (Pham, *et al.*, 2019; Pecorini *et al.*, 2020; Al-Shafei *et al.*, 2022).

Table 9. Physicochemical and biochemical properties of *Milicia excelsa* bio-oil yield at optimum temperature (550 °C)

Properties	Biomass/catalyst ratio	Bio-oil composition (%)		
		100/0	80/20	60/40
Ultimate Analysis	C	60.96	80.50	80.76
	H	10.90	12.33	10.45
	N	0.35	0.86	0.65
	S	0.01	0.06	0.1
	O	27.78	6.25	8.04
Proximate Analysis	Moisture Content	8.56	4.80	2.82
	Volatile matter	74.80	15.32	15.70
	Fixed Carbon	14.86	76.68	78.98
	Ash	1.78	3.20	2.50
Heating value	HHV (MJ/kg)	25.78	33.04	31.55
Molar Ratio	H/C	2.15	1.84	1.55
	O/C	0.34	0.06	0.08
	(O + N)/C	0.35	0.07	0.08
Biochemical Analysis	Lignin	5.56	4.16	4.15
	Cellulose	41.04	43.50	43.56
	Hemicellulose	50.55	51.77	52.09

The oxygen content in bio-oil decreased with increase in catalyst, for favorable heating value enhancement. The biochemical analysis revealed the percentage of lignin (Li), cellulose (Ce) and hemicellulose (He) of bio-oil produced from *Me* (Table 9). Higher compositional percentage of Cellulose

and hemicellulose in catalytic bio-oil indicates increased presence of aromatic hydrocarbons compared to non-catalytic bio-oil. Both cellulose and hemicellulose consist of sugar monomers which make the duo suitable for bio-fuel production (Lin *et al.*, 2015; Chukwuneke *et al.*, 2019; Sarkar & Wang, 2020; Oyekale & John, 2023). The non-catalytic bio-oil had the lowest HHV of 25.78 MJ/kg, while catalytic bio-oil HHV were 33.04 and 31.55 MJ/kg at biomass/catalyst ratios of 80/20 and 60/40, respectively. This is near to the energy value of conventional diesel, which is between the range of 39.57 and 41.33 MJ/kg (Mujtaba *et al.*, 2023).

4. CONCLUSION

The impact of temperature and green produced zeolite-Y catalyst on the yields and quality of bio-oil production was investigated in this study. According to the findings, the main chemical components of the bio-oil yield generated under both catalytic and non-catalytic conditions were phenols and hydrocarbons. The bio-oil was significantly improved at optimum pyrolysis temperature of 550 °C with biomass/catalyst ratios loading of 100:0, 80:20 and 60:40 of *Milicia excelsa*. The catalytic bio-oils at 60/40 ratio has highest phenol percentage (59.59%) with highest higher heating value (HHV) of the catalytic bio-oil (33.04 MJ/kg) at 80/20 ratio, which indicated the catalytic pyrolysis led to bio-oil with higher energy content. Catalyst reduced the bio-oil acidity to 6.4 -7.6 and flash point (79 °C) at optimum pyrolysis temperature of 550 °C in comparison to petroleum diesel with acidity of 5.5 - 8 and flash point (75 °C). The catalytic pyrolysis of *Milicia excelsa* produced enhance bio-oil with high fuel quality and value-added compounds.

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