



Catalytic Pyrolysis of Malaysian Tea Waste over Molybdenum Modified Zeolite for Bio-Oil Production

Muhammad Hadif Haidir¹, Vekes Balasundram^{1,*}, Norazana Ibrahim², Ruzinah Isha³, Suchithra Thangalazhy Gopakumar⁴, Yazid Bindar⁵, Le Kim Hoang Pham⁶, Suan Shi⁷, Suneerat Fukuda⁸

- ¹ Malaysia-Japan International Institute of Technology (MJIIT), Universiti Teknologi Malaysia, Jalan Sultan Yahya Petra, 54100 Kuala Lumpur, Malaysia
- ² Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia
- ³ Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, Lebuhr Persiaran Tun Khalil Yaakob, 26300 Kuantan, Pahang, Malaysia
- ⁴ Faculty of Science and Engineering, University of Nottingham Malaysia, Jalan Broga, 43500 Semenyih, Selangor Darul Ehsan, Malaysia
- ⁵ Fakultas Teknologi Industri, Institut Teknologi Bandung, Kota Bandung, Jawa Barat 40132, Indonesia
- ⁶ Institute of Applied Technology and Sustainable Development, Nguyen Tat Thanh University, Ho Chi Minh City, 755414, Vietnam
- ⁷ Engineering Laboratory for Agro Biomass Recycling & Valorizing, College of Engineering, China Agricultural University, Beijing 100083, China
- ⁸ Energy Technology & Management, King Mongkut's University of Technology Thonburi (KMUTT), 126 Pracha-uthit Rd., Tungkrui, Bangkok 10140, Thailand

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ABSTRACT

In the current era, the increasing population has raised energy needs and heavy reliance on fossil fuels has led to greenhouse gas emissions. The abundance of biomass in Malaysia, such as tea waste, is poorly managed, contributing to environmental issues. Thus, the main goal of this research is to investigate the influence of Molybdenum-modified Hydrogen-exchanged Zeolite Socony Mobil-5 (Mo/HZSM-5) on the pyrolysis of Malaysian tea waste at varied temperatures from 400°C to 600°C with 50°C intervals to produce bio-oil via fixed-bed reactor. The catalyst-to-tea waste mass ratio was constant at 1:1 for all the investigated samples. The HZSM-5 was used as a support at 99 wt.%, while the balance 1 wt.% was impregnated with molybdenum (Mo) via the incipient wetness impregnation method. Among the investigated samples, the highest bio-oil produced was at 600°C (13.97 wt.%). Surprisingly, adding Mo/HZSM-5 did not increase the bio-oil yield, which is 4.50 wt.% at 600°C. However, the composition of hydrocarbons in bio-oil from catalytic pyrolysis is higher (18.16 %) than non-catalytic (9.73 %) at 500°C. Furthermore, with Mo/HZSM-5, the total oxygenated compound presences are lower (51.77 %) at 550°C than without catalyst (62.16 %). Therefore, pyrolysis using Mo/HZSM-5 as a catalyst has proved to increase the amount of hydrocarbon in bio-oil while lighter hydrocarbons (C₉ to C₁₅) were enhanced at lower temperatures (400°C) by the cracking of larger hydrocarbon molecules.

* Corresponding author.

E-mail address: hadif01@graduate.utm.my

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1. Introduction

The energy demand has increased concurrently with the increase in population [8]. The Shift Project [1] reports that the world is expected to suffer from oil depletion by 2030, as the total volume produced will likely shrink by up to nearly 8 %. This illustrates the rapid depletion of energy resources due to environmental changes that have prompted the search for sustainable and clean energy. Furthermore, Woodard *et al.*, [2] mentioned that the extensive dependence on fossil fuels for energy production has contributed to global environmental challenges, such as climate change. The emission of greenhouse gases (GHGs), particularly carbon dioxide (CO₂), exceeds the earth's natural capacity to maintain carbon balance. Therefore, it is crucial to explore sustainable methods of energy production.

Not only the energy issue but also the generation of waste has increased due to the population. As reported by the International Trade Administration [3], Malaysia generates a significant amount of waste, producing approximately 30,000 tonnes of municipal solid waste (MSW) daily, which equates to a per capita daily waste volume of 1.17 kg per household. The amount of waste disposed of has steadily increased alongside the country's population growth, with most waste deposited in landfills. Moreover, MSW consists of paper, food, plastic mixed organic waste, wood and other materials.

According to Kasim Mansur [5], tea production in Malaysia is a continuous process, with harvesting occurring every three to four weeks to coincide with the growth of new shoots. Tunklová *et al.*, [4] indicated that, tea waste (TW) is part of food waste, consisting of lignocellulosic material. Consequently, Liew [5] also reported that Malaysian TW yearly output amounts to approximately 3.6 million kilograms, with a substantial proportion being supplied by BOH, followed by the plantations of Bharat Tea and Sabah Tea. As tea production increases annually, TW is also constantly produced. Thus, the amount of TW can be reduced *via* the waste-to-energy (WTE) technology.

According to Kristiina *et al.*, [6], pyrolysis is a promising method for converting biomass waste into bio-oil. However, Li *et al.*, [7] mentioned that pyrolysis alone is insufficient for producing high-grade bio-oil from biomass. Significant studies have been made over the past decade to find suitable catalysts. Thus, a metal-modified Hydrogen-exchanged Zeolite Socony Mobil-5 (HSZM-5) catalyst in the pyrolysis of biomass waste enhances the selectivity of desired products, reduces the energy needed for pyrolysis and improves catalytic cracking while minimizing coke formation. Therefore, the objective of this research was to investigate the effect of molybdenum-modified HZSM-5 catalyst for catalytic pyrolysis of TW at varied temperatures aiming to produce bio-oil rich in hydrocarbons that meet high-quality fuel standards and can compete with conventional fuels.

2. Methodology

2.1 Tea Waste Preparation

In this study, TW was obtained from various restaurants around Taman Medan, Petaling Jaya, Selangor. The TW was filtered from the tea bag and collected in the wet state. Next, it was washed with distilled water and stirred for 3 hours. Then, it was filtered to remove water and eventually dried in a laboratory oven at 110°C for about 12 hours until the weight remained constant. Lastly, the dried TW was stored in closed containers to avoid exposure to moisture. Meanwhile, the preparation and characterization of the Mo/HZSM-5 were reported in our previous work [23].

2.2 Pyrolysis Process

The effect of molybdenum-modified HZSM-5 catalyst on the pyrolysis of Malaysian TW was investigated *via* laboratory-scale fixed bed reactor. The setup included a reactor with a fixed bed, a tubular furnace for heating, a condenser system with a filtering flask immersed in an ice bath, a gas bag and nitrogen (N₂) source as an inert gas as shown in Figure 1. Before each experiment, 2 g of Mo/HZSM-5 and 2 g of TW sample were mixed thoroughly and loaded onto the fixed bed, maintaining a catalyst-to-biomass ratio of 1:1 by mass. Inlet and outlet connections were securely sealed to prevent leakage during experiments. N₂ gas was used to purge the reactor to achieve an inert environment, with a flow rate of 100 mL/min. The experiments were conducted under atmospheric pressure (1 atm), with the tubular furnace heated to specific temperatures ranging from 400 to 600 °C in 50 °C intervals, controlled *via* an electrical panel. During reactions, N₂ gas carried reactive substances downwards through the reactor and into the condenser (filtering flask) immersed in an ice bath, where gaseous products condensed into liquid pyrolysis oil. This oil was subsequently analyzed to determine its chemical composition. Meanwhile, the non-condensable vapours were collected in the gas bag. After each experiment, the reactor was disassembled, and its contents were separated. To ensure accuracy, each test was repeated at least twice.

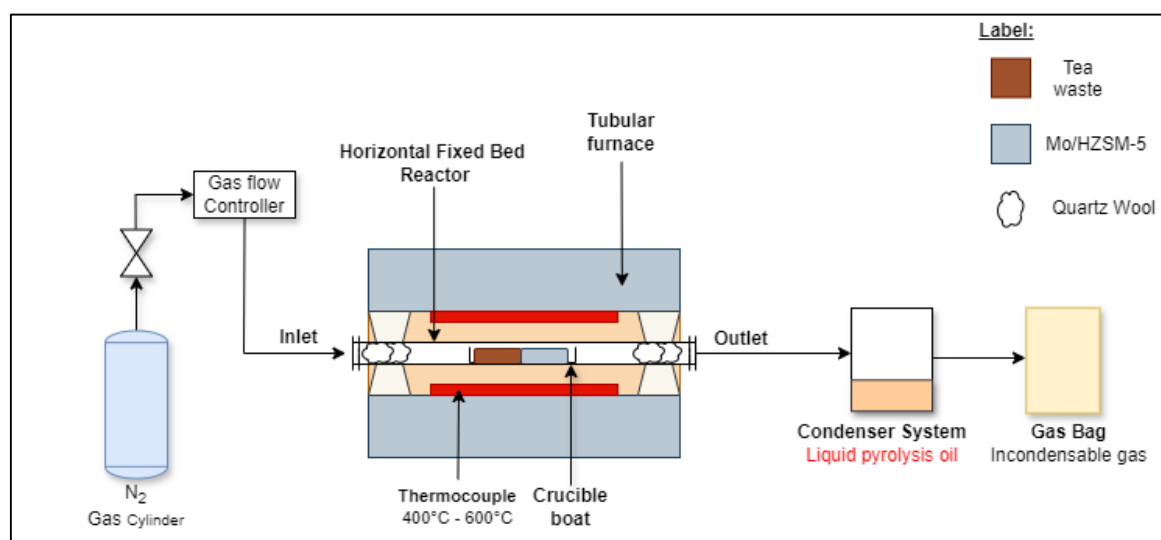


Fig. 1. Schematic diagram of fixed bed pyrolysis set-up

2.3 Bio-Oil Analysis

Bio-oil from the Malaysian TW was analyzed using gas chromatography-mass spectrometry (GC/MS) with an Agilent 6890N series GC with a 5973N Inert MS detector and 7683 Injector. The bio-oil was then diluted 1:1 with methanol and was separated on an HP-5 MS capillary column (30 m length, 0.25 mm diameter and 0.25 µm thickness). A 1 µL sample was injected at 250°C in split mode (100:1). Helium (99.999 % purity) was the carrier gas at 1.2 mL/min. The column temperature started at 40°C, increased to 100°C at 20°C/min, then to 250°C at 5°C/min. The mass spectrometer, in electron impact (EI) mode at 70 eV and 250°C, scanned 30-500 m/z. Chromatographic peaks were identified using the NIST database to determine the bio-oil's composition.

3. Results

3.1 Pyrolysis of Malaysian Tea Waste

3.1.1 Product yields

Figure 2 shows the distribution of products from the pyrolysis of TW, which are oil, gas and char at various temperatures ranging from 400 to 600 °C in 50 °C intervals. The results indicate an increasing pattern for the oil yield, clearly showing that the pyrolysis temperature significantly impacts the oil yield. Overall, the highest oil yield (13.97 wt%) was discovered at 600 °C followed by 550 °C (11.50 wt%), 500 °C (10.30 wt%), 450 °C (8.75 wt%) and 400 °C (7.75 wt%). Similarly, Daniel *et al.*, [9] found that pyrolysis of biomass at 600 °C maximized bio-oil production, achieving a high yield of 55.3 wt%. This substantial bio-oil yield is attributed to the high lignin content in the composition of biomass. Therefore, the composition of the TW allows it to produce higher oil yield at higher temperatures.

Not only oil but also gas and char yields were observed. Gas yield production is notably higher, ranging from 48.75 to 56.75 wt%, compared to oil and char yields. Figure 2 illustrates an increasing trend in gas yield from 400 to 550 °C; however, it decreases at 600 °C (55.11 wt%), likely due to the transition from slow to fast pyrolysis. According to Tan *et al.*, [10], rapid heating and increasing temperatures in fast pyrolysis can enhance bio-oil production relative to gases. Consequently, when the temperature exceeds 600 °C, gas yield decreases while bio-oil yield increases, as Figure 2 also shows the highest oil yield achieved at 600 °C. For char yield, as depicted in Figure 2, it was consistently decreased from 400 to 600 °C, ranging from 43.50 to 30.92 wt%, likely due to higher temperatures causing extensive decomposition of organic matter. Alperay *et al.*, [11] found that an increase in pyrolysis temperature to 600 °C led to a decrease in biochar yield (30.60 wt%), while synthesis gas yield increased (56.64 wt%). Thus, higher temperatures result in reduced char yield due to the degradation of organic matter.

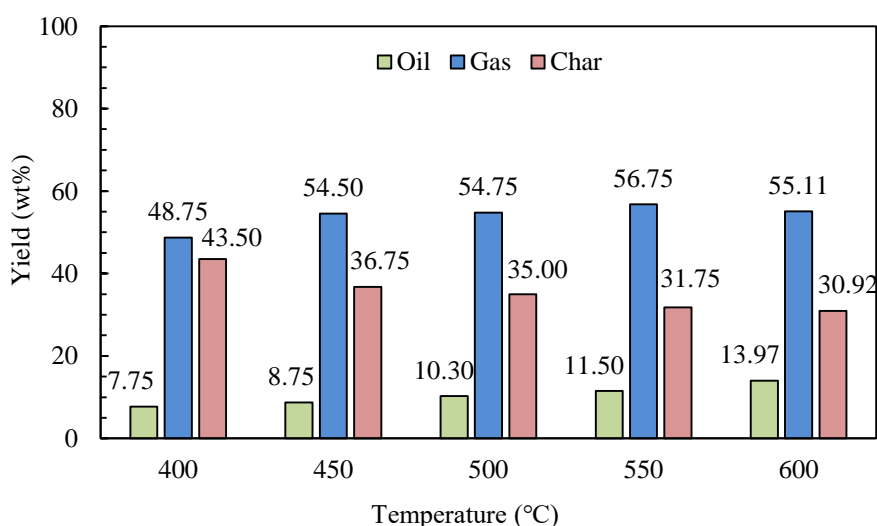


Fig. 2. The yield of pyrolysis of tea waste

3.1.2 Organic compositions of bio-oil

Table 1 illustrates the organic compositions (%) in pyrolysis oil derived from the pyrolysis of TW at temperatures ranging from 400 to 600 °C. The results were categorized according to the different temperatures. The chemical compounds produced were classified into hydrocarbons, phenols,

ketones, aldehydes, esters, alcohols, acids, nitrogenated compounds (N-compounds) and others (such as caffeine, furan and anhydrous sugar).

The highest hydrocarbon content in the pyrolysis oil was observed at 500 °C (9.73 %), followed by 450 °C (8.71 %), 600 °C (8.53 %), 550 °C (8.07 %) and 400 °C (7.17 %). In this study, hydrocarbons were consistently below 10 % at all temperatures, which is lower compared to other biomass types like softwood pellets that can generate hydrocarbons in the range of 40 to 55.7 % at 500 °C *via* pyrolysis, as reported by Zhang *et al.*, [12]. Higher temperatures during pyrolysis can lead to secondary reactions such as cracking and reforming of primary vapours, resulting in less liquid pyrolysis oil and potentially lower hydrocarbon content due to the formation of non-condensable gases and char. Similarly, Nagaraja [13] noted that increasing temperatures enhance the thermal decomposition rate, leading to higher gas production, which could decrease the hydrocarbon content in pyrolysis oil at higher temperatures.

Table 1
Yield of pyrolysis of tea waste

Compounds	Temperature (°C)				
	400	450	500	550	600
Hydrocarbons	7.17	8.71	9.73	8.07	8.53
C ₉ - C ₁₄	0.83	2.15	2.56	3.89	1.20
C ₁₅ - C ₂₃	6.34	6.56	6.66	4.18	6.98
Phenols	22.38	26.13	26.86	31.41	19.52
Ketones	3.15	6.09	4.15	2.62	5.27
Aldehydes	-	0.32	0.98	-	-
Alcohols	4.56	3.84	5.55	4.77	7.69
Acids	24.16	21.27	15.76	22.89	20.74
Ester	0.59	0.32	-	0.47	0.66
N-Compounds	7.63	5.85	6.96	6.89	3.70
Others	30.36	27.47	30.01	22.88	33.89

Based on Table 1, the production of hydrocarbons was more favourable at temperatures between 400 °C and 500 °C. Detailed analysis reveals that heavy hydrocarbons (C₁₅ - C₂₃) were produced in the pyrolysis oil at these temperature ranges. This occurs because the endothermic nature of the cracking process which requires energy, prevents the effective breakdown into light hydrocarbons (< C₁₅). The cracking process demands additional energy to break the bonds within larger hydrocarbon molecules weaker than the newly formed bonds. However at temperatures above 500 °C, the cracking process intensifies, forming much lighter hydrocarbons (C₉ - C₁₄). Consequently, larger hydrocarbon molecules in the feedstock, such as long-chain alkanes or aromatics, are more likely to convert into smaller hydrocarbons at temperatures above 500 °C [14].

Lastly, pyrolysis of TW also produces oil rich in oxygenated compounds, with a significant phenol content (36.32 %) at higher temperatures due to lignin decomposition. Also, ketones and aldehydes were present, with ketones peaking at 450 °C (6.09 %) due to their volatility, while aldehydes were observed at 450 °C (0.32 %) and 500 °C (0.98 %), that were likely from the hemicellulose breakdown. Furthermore, alcohols in the pyrolysis oil vary with temperature which peaked at 600 °C (7.69 %). This could be due to the influence of sugar content in tea leaves. In addition, acids were most abundant at lower temperatures but fluctuated at higher ones due to molecular instability. Esters formed at all temperatures except at 500 °C, with the highest at 600 °C (0.67 %) due to sufficient alcohol and acid presence. Nitrogen compounds remain stable between 400 °C and 550 °C but decrease significantly at 600 °C. Other compounds, like caffeine, furans and anhydrosugars, were also present, with caffeine levels reflecting tea's natural content.

3.2 Catalytic Pyrolysis of Malaysian Tea Waste

3.2.1 Product yields

Figure 3 illustrates the distribution of products from the catalytic pyrolysis of TW at various temperatures ranging from 400 to 600 °C in 50 °C intervals. The trend in the oil yield graph showed fluctuations, with the highest oil yield observed at 600 °C (4.50 wt%), followed by 500 °C (4.00 wt%), 550 °C (3.00 wt%), 400 °C (1.5 wt%) and 450 °C (1.00 wt%). At 500 °C, the increase in yield suggests optimal conditions for cracking and condensation processes, enhancing oil production. However, the yield decreased at 550 °C, likely due to secondary oil cracking into gas. The highest yield at 600 °C may be attributed to the ability of the Mo/HZSM-5 catalyst to enhance volatilization processes, although it may still convert some oil into gas at high temperatures. According to Rehan *et al.*, [15], zeolite catalysts can synergistically reduce oxygen content by breaking down heavier hydrocarbons into lighter ones, thereby increasing gas yield. Compared to pyrolysis without a catalyst, catalytic pyrolysis shows a lower oil yield (7.75 - 13.97 wt%). This could be due to the strong acidic properties of Mo/HZSM-5 which facilitate the breakdown of heavier hydrocarbons into lighter ones. Similarly, Daligaux *et al.*, [16] observed that catalytic pyrolysis of biomass results in a higher gas yield (63.50 wt%) compared to thermal pyrolysis (13.00 wt%). This is because some hydrocarbons that would form part of the liquid oil were instead converted into non-condensable gases. Thus, it is evident that Mo/HZSM-5 can break down heavier hydrocarbons into lighter ones, but at higher temperatures, lighter hydrocarbons escape as gas instead of forming liquid oil.

Moreover, gas yield shows a significant increase with rising temperatures. As illustrated in Figure 3, the highest gas yield was recorded at 600 °C (73.00 wt%), followed by 550 °C (72.50 wt%), 500 °C (68.50 wt%), 450 °C (67.00 wt%) and 400 °C (57.50 wt%). This increase can be attributed to the ability of Mo/HZSM-5 to enhance cracking at lower temperatures by reducing the activation energy. At higher temperatures, it effectively converts more products into the gaseous phase. Similarly, Syamsiro *et al.*, [17] observed that catalytic acid sites promote cracking reactions, resulting in catalytic pyrolysis producing lighter products than thermal pyrolysis, thereby increasing the gaseous fraction and reducing the liquid fraction. In comparison to non-catalytic pyrolysis, the use of a catalyst in the pyrolysis of TW results in a higher gas yield.

Additionally, Figure 3 shows that char yield exhibits an inverse relationship with temperature, consistently decreasing as the temperature rises. The highest char yield was recorded at 400 °C (41.00 wt%), followed by 450 °C (32.00 wt%), 500 °C (27.50 wt%), 550 °C (24.50 wt%) and 600 °C (22.50 wt%). This decrease is likely due to the complete degradation of the TW, leaving less solid residue. Both pyrolysis and catalytic pyrolysis display the same decreasing trend in char yield, as the increasing temperature promotes the cracking of more valuable products such as oil and gas rather than char. According to Li *et al.*, [18], the presence of a zeolite catalyst significantly increases the gas yield at a pyrolysis temperature of 700 °C, suggesting that the catalyst shifts the product distribution from char towards more gaseous products at elevated temperatures. Therefore, it is evident that adding a catalyst to the pyrolysis of TW enhances the production of more valuable products.

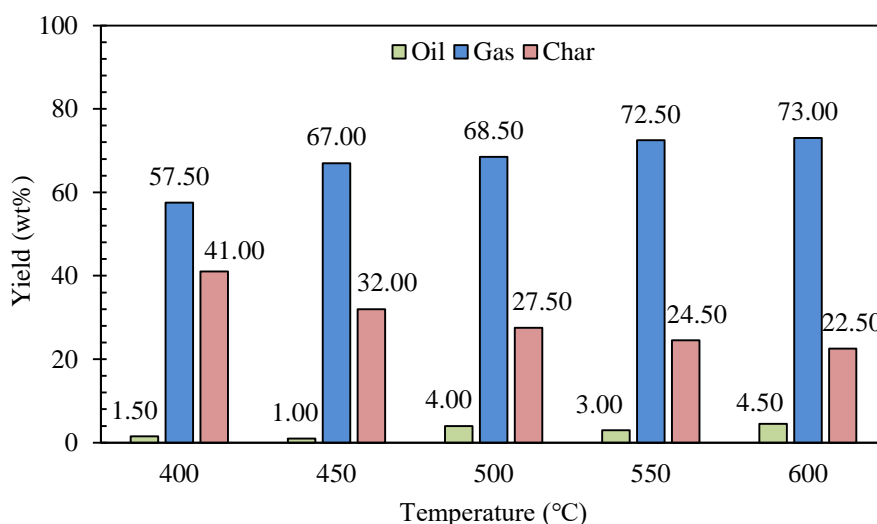


Fig. 3. The yield of pyrolysis of tea waste

3.2.2 Organic compositions of bio-oil

Table 2 shows the organic compositions (%) in pyrolysis oil from catalytic pyrolysis of TW over Mo/HZSM-5 at 400 to 600 °C. Similar compounds in pyrolysis oil from pyrolysis of TW were produced from catalytic pyrolysis, such as hydrocarbons, phenols, ketones, aldehydes, ester, alcohols, acids, N-compounds and others such as caffeine, furan and anhydrous sugar. The highest hydrocarbon content was observed at 500 °C (18.16 %), followed by 450 °C (17.02%), 550 °C (14.80 %), 600 °C (13.52 %) and 400 °C (7.65 %). At 500 °C, Mo/HZSM-5 played a crucial role in enhancing deoxygenation reactions, converting oxygenated compounds in the bio-oil into hydrocarbons. Rizki *et al.*, [19] noted that molybdenum-based catalysts are highly effective in hydro-deoxygenating biomass pyrolysis vapours, resulting in increased hydrocarbon production and reduced oxygen content.

Table 2
Yield of catalytic pyrolysis of Malaysian tea waste

Compounds	Temperature (°C)				
	400	450	500	550	600
Hydrocarbons	7.65	17.02	18.16	14.80	13.52
C ₉ to C ₁₄	4.01	9.64	5.97	4.72	3.49
C ₁₅ to C ₂₈	3.64	7.38	12.19	10.08	10.03
Phenols	23.92	27.9	33.86	29.32	36.32
Ketones	3.54	2.94	1.81	3.12	3.44
Aldehydes	-	0.91	1.08	-	0.71
Alcohols	2.98	4.23	2.89	6.98	2.16
Acids	14.22	10.91	7.07	11.63	10.06
Ester	0.63	1.27	2.87	0.72	3.38
N-Compounds	8.43	5.52	9.20	8.28	7.54
Others	38.63	29.3	23.06	25.15	22.87

Interestingly, increasing the temperature beyond 500 °C does not result in a further increase in hydrocarbon content despite the presence of Mo/HZSM-5. This could be attributed to an increased

likelihood of coke formation at higher temperatures, which can block active sites on the catalyst and reduce its effectiveness in converting oxygenated compounds into hydrocarbons. Kosinov *et al.*, [20] observed that coke formation over Mo/zeolite catalysts occurred at elevated temperatures. Therefore even with the presence of Mo/HZSM-5, the hydrocarbon contents decreased at temperatures above 500 °C.

Temperatures of 450 °C and 500 °C favour higher production of hydrocarbons. Compared to non-catalytic pyrolysis oil, catalytic pyrolysis with Mo/HZSM-5 significantly increases hydrocarbon production, except at 400 °C (Table 2). This indicates that Mo/HZSM-5 plays a crucial role in enhancing hydrocarbons in bio-oil. Lighter hydrocarbons (C₉ - C₁₅) were produced at lower temperatures (450 °C), whereas only temperatures higher than 500 °C produce lighter hydrocarbons (C₉ - C₁₄) in non-catalytic pyrolysis oil. This difference underscores the catalyst's efficiency in converting large lignocellulosic molecules into smaller hydrocarbon fractions at lower temperatures. Rahman *et al.*, [21] noted that catalysts can initiate cracking reactions, breaking down large molecules within pyrolysis products, which aligns with the observed outcomes.

Based on Table 2, temperatures 450 °C and 500 °C are more favoured with high production of hydrocarbons. Compared with non-catalytic pyrolysis oil, the amount of hydrocarbon produced was much higher except at 400 °C. Thus, it can be concluded that the Mo/HZSM-5 played a significant role in enhancing the hydrocarbons in pyrolysis oil. It can be observed that the lighter hydrocarbons of C₉ - C₁₅ were produced at lower temperatures (450 °C). In comparison with the non-catalytic pyrolysis oil, it can be observed that only at higher temperatures than 500 °C it can produce lighter hydrocarbons (C₉ - C₁₄). This is because catalysts are capable of efficiently converting large lignocellulosic molecules into smaller hydrocarbon fractions at lower temperatures, leading to the observed outcome. Imran *et al.*, [22] reported that adding a catalyst can induce cracking reactions, leading to the breakdown of large molecules within pyrolysis products. Therefore, this process yields bio-oils that exhibit lighter viscosity and contain a higher concentration of valuable compounds such as hydrocarbons and phenols.

Lastly, at higher temperatures (above 500 °C) phenol production was enhanced at 600 °C yielding the most (36.32 %). This is due to catalysts accelerates the lignin decomposition and promote deoxygenation reactions, which converts biomass into aromatic compounds. Moreover, ketone production peaks at 400 °C (3.54 %) and is generally lower than non-catalytic pyrolysis, due to catalysts that reduce unwanted compounds like aldehydes and ketones. Next, alcohol yields peak at 550 °C (6.98 %) with catalysts, which breakdowns complex molecules at lower temperatures. Also, acid production was lower with catalysts, peaking at 400 °C (14.22 %) compared to pyrolysis without catalysts. Ester production was higher with catalysts at 600 °C (3.38 %) that was due to enhanced dehydration reactions. On the other hand, nitrogen compounds peaked at 500 °C (9.20 %) in catalytic pyrolysis, which was slightly higher than non-catalytic methods. This was likely due to TW's high nitrogen content. Catalytic pyrolysis also produced caffeine, furan and anhydrosugars, with caffeine levels potentially preserved by the catalysts.

4. Conclusions

In conclusion, this study evaluated the effect of molybdenum-modified HZSM-5 catalyst for catalytic pyrolysis of TW at varied temperatures, addressing energy recovery, carbon neutrality and effective biomass waste management by producing valuable energy products. Optimal hydrocarbon yields (18.16 %) were achieved at 500 °C due to effective deoxygenation and cracking reactions facilitated by the catalyst. Furthermore, lower temperatures (400 to 500 °C) favoured higher hydrocarbon content (7.17 - 9.73 %) compared to non-catalytic pyrolysis, while temperatures above

500 °C reduced hydrocarbon yields due to intensified cracking. Therefore, these findings underscore the catalyst's role in promoting cleaner liquid hydrocarbon production from biomass.

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