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Production and characterization of bio-oil derived from microwave-assisted pyrolysis of *Dodonaea viscosa*, employing HZSM-5 and titanium dioxide catalysts

Marwa I. Abbas[1,2], Atheer M. Al-Yaqoobi[1], Haider Abdulkareem Al-Jendeel[1*]

[1] Department of Chemical Engineering, College of Engineering, University of Baghdad, Aljadria, Baghdad, 10071, Iraq
[2] Biochemical Engineering Department, Al-Khwarizmi College of Engineering, University of Baghdad, Baghdad 47024, Iraq
* Corresponding author's e-mail: haider.aljendeel@coeng.uobaghdad.edu.iq

ABSTRACT

The current study has focused on microwave-assisted pyrolysis of *Dodonaea viscosa* for producing bio-oil using two different catalyst types, HZSM-5 and TiO_2. The experiment included varying the microwave power (500–600 W), reaction time (5–20 minutes), and weight percentage of catalyst (20, 30, and 50 wt%) to evaluate the effects of these variables on the product yield and properties bio-oil. The bio-oil was characterized through comprehensive methods, including GC–MS and physical/property measurements, including viscosity, pH, and higher heating values (HHV). The results show that the catalyst significantly affects both the quantity and quality of the bio-oil generated. TiO_2 showed the best results for bio-oil yield (32.42 wt%) under 600 W and 20 minutes, indicating that it can facilitate controlled thermal decomposition while minimizing excessive secondary reactions. Conversely, HZSM-5 yielded a much lower maximum yield (25.7 wt%) at 500 W; however, it greatly improved the quality of the bio-oil. The HHV was increased to 25.4 MJ/kg when HZSM-5 was used instead of TiO_2 (21 MJ/kg). The GC–MS analysis demonstrated that HZSM-5 promoted deoxygenation/aromatization reactions, reactions leading to higher aromatic (21.3%) and phenol (21.57%) and lower oxygenated compounds such as acids (12.9%). On the contrary, TiO_2 had a larger proportion of ketones (%18.2) and acids (%17.1); these data reflect a moderate catalytic activity for TiO_2 and less potential for upgrading bio-oil. In general, this study indicates that TiO_2 is more efficient at increasing bio-oil yields, whereas HZSM-5 is better at enhancing bio-oil quality.

Keywords: microwave catalytic pyrolysis, *Dodonaea viscosa*, HZSM-5, TiO_2, bio-oil.

INTRODUCTION

The increasing cost of energy and global demand, along with the environmental problems caused by consuming fossil fuels, have stimulated the pursuit of new sustainable and renewable energy resources (Holechek et al., 2022). The reduction in fossil fuel reserves and rising cost of energy will negatively affect the world's economy in the following decades. Energy costs are predicted to continue to climb, and it is estimated that fuel consumption will increase dramatically (Rueangsan et al., 2025). Biomass is one example of an alternative energy resource that has garnered much attention because of its renewability, abundance, and lower environmental impacts compared to traditional fossil fuels (Nishu et al., 2020).

Biomass is being explored as a promising feedstock for the development of biofuels and value-added products through thermochemical conversion processes such as gasification, hydrothermal liquefaction, and pyrolysis (Zhang et al., 2019). Pyrolysis has been developed as a versatile and efficient method to convert solid biomass to bio-oils, biochars, and non-condensable gases through thermal decomposition under oxygen-free conditions (Abd and AL-Yaqoobi, 2025). Specifically, fast pyrolysis is well known for achieving maximum bio-oil yields and generating

a variety of liquid products with potential applications (Bridgwater, 2012).

The use of bio-oil as a substitute for petroleum-based fuels or as a feedstock for chemical manufacturing is becoming increasingly popular. Despite interest in using bio-oil as a replacement for petroleum-based fuels or as a feedstock for chemical manufacturing, several limitations restrict its direct use. These features include a high oxygen content (35–40 wt%), which leads to acidic properties; high viscosity; low calorific value; and limited thermal stability (Dabros et al., 2018; Rezaei et al., 2014). Many different functional groups of oxygen in bio-oil, including carboxylic acids, ketones, aldehydes, phenols, and furans, lead to storage instability and degradation during combustion. Consequently, upgrading processes are necessary to produce a bio-oil that satisfies the required specifications (Balasundram et al., 2022)

Pyrolytic conversion can be enhanced catalytically to yield superior bio-oil with enhanced physical and chemical characteristics using zeolites (Wang et al., 2017). Catalytic pyrolysis involves three main types of reactions: deoxygenation, cracking, and aromatization, which reduce oxygen in the bio-oil, create hydrocarbons, and increase its energy density (Rahman et al., 2018a). Catalysts possessing Brønsted acidity have shown excellent activity for zeolites. In particular, HZSM-5 displays strong Brønsted acidity and shape selectivity, offering benefits for converting oxygenated species into aromatic hydrocarbons. Also, its Brønsted acidity encourages secondary reactions (cracking and polymerization), which leads to lower bio-oil yields and higher gaseous products (Dada et al., 2023; Liu et al., 2020; Shi et al., 2017).

Metal oxides, like titanium dioxide, show milder catalytic activity and redox behavior compared to zeolite catalysts. Metal oxide catalysts allow partial deoxygenation while keeping high bio-oil yields (Sun et al., 2025). Since they are resistant to excessive secondary reactions, they provide a more balanced route between product yield and quality (Qiu et al., 2024).

Recently microwave-assisted pyrolysis has become a novel approach to pyrolysis due to the specific way it heats materials. Microwave heating provides internal heating throughout the bulk biomass via dipolar rotation and ionic conduction. Unlike traditional heating methods, which cause temperature gradients and inefficient heat transfer, microwave heating offers uniform volumetric heating which boosts heat transfer efficiency, leading to the formation of reactive intermediate species like free radicals. Free radicals determine how products will distribute during pyrolysis (Ahmad et al., 2026).

Although little research has examined the synergistic effect of microwave-assisted pyrolysis combined with different catalysts on *Dodonaea viscosa*, a non-food lignocellulosic biomass native to arid regions worldwide. (Hamadi, 2017; Shakir and Al-Yaqoobi, 2025), This biomass offers many advantages, such as rapid growth, adaptability to harsh climate conditions, and sustainability as a feedstock (Liu et al., 2022).

Thus, the purpose of this study was to investigate the effectiveness of HZSM-5 and TiO_2 catalysts in the microwave-assisted pyrolysis of *Dodonaea viscosa*. In addition, the physicochemical properties of the produced bio-oils were evaluated, including as viscosity, density, pH, HHV values, and GC-MS analysis, as well as the influence of operational parameters such as microwave power, reaction time, and catalyst loading were also examined.

MATERIALS AND METHOD

Materials

The *Dodonaea viscosa* branches used in this search were taken from the campus of the University of Baghdad in Baghdad, Iraq. The branches were cleaned by washing them first in tap water and then in distilled water, after which they were sun-dried until the excessive moisture was removed for 48 hours. After drying, the branches were crushed with a grinder to produce fine powder. The fine powder was separated through sieving to isolate a uniform particle size of approximately 500 μm. The uniformly sized particles were stored in a desiccator. In this study two different catalysts were used: TiO_2 (titanium dioxide or titanium (IV) oxide) 98.0% pure (from New Delhi-030446, INDIA) and HZSM-5, with a surface area of 350 m^2/g, an SiO_2/Al_2O_3 ratio of 46, a particle size of 0.60 μm, and 95% crystallinity (from Henan Province, China).

Material preparation

Catalyst HZSM-5 and TiO_2 were combined at concentrations of 20, 30, and 50%, along with raw material weights, to create sample types. TiO_2

catalyst was used as powder, while HZSM-5 catalyst was formed into pellets; this was achieved through the use of a Mini Screw Extruder (Caleva, England) and a granulator to form a catalyst pellet with a 0.12 cm diameter, as illustrated in Figure 1, so they would be easy to remove from the biochar. 25% bentonite was added to 100 g of powdered catalyst as a binder to achieve an extrudate catalyst. (Al-Taweel et al., 2025)After drying, the catalysts were calcined at 600 °C and 100 °C for 5 hours, respectively (Rajwanti and Tyagi, 2022).

Method

The experimental design of each experiment was similar to the one illustrated in Figure 2. Pyrolysis was performed using a microwave oven (DOMO Solo, 30L, China) with a maximum power of 900 W. two different pyrolysis powers were tested (500 and 600 W). The reactor of a 150 ml conical flask having a flat base and a socket diameter of 29/32. It had an inlet tube through which N_2 gas, as an inert gas (99% pure), could be introduced from the outside. The outlet tube carried the pyrolysis vapors to a condensate collection unit consisting of two cylindrical glass tubes placed in an ice-bath at a temperature -17 °C. Vapors that did not get condensed out were released to the atmosphere. Biochar and bio-oil were produced during the pyrolysis. After the pyrolysis reaction, the resulting biochar was collected, while the generated vapors were condensed into bio-oil. The yields of bio-oil, biochar, and biogas were evaluated using Equations 1, 2 and 3, respectively, and were expressed based on the mass of the reactants, all the results were based on the mass of reactants.

$$Yield\ of\ Bio-oil\ (\%) = = \frac{Mass\ bio-of\ oil}{Mass\ of\ feedstock} \times 100 \quad (1)$$

$$Yield\ of\ biochar\ (\%) = \frac{mass\ of\ biochar}{Mass\ of\ feedstock} \times 100 \quad (2)$$

$$Yield\ of\ biogas\ (\%) = 100 - - (yield\ biochar + yield\ Bio - Oil) \quad (3)$$

The reactor was first filled with feedstock in which the feedstock mixed with 20 g of catalyst at weight percentages of 20, 30, and 50%, with biomass particle sizes of 500 µm. To establish an oxygen-free atmosphere, before the start of the experiment, 10 minutes of inert nitrogen gas was supplied into the device at a flow rate of 1 L/min. The microwave was thereafter engaged at a designated power level.

Products analysis

The physical and chemical properties of the generated bio-oil were studied by analyzing them in terms of their application possibilities. To study the physical parameters of the biooil, fixed standards were utilized for determining its properties, which included viscosity, acidity, calorific value, and density. The bio-oil's viscosity at 40 °C was

Figure 1. HZSM-5 catalyst

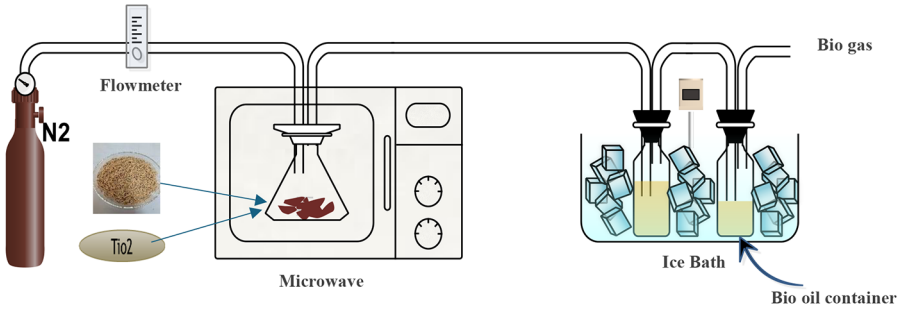

Figure 2. Schematic diagram of the catalyzed bio-oil preparation steps

measured with a U-tube viscometer, while its density at 25 °C was determined using a pycnometer. The acidity was measured using a pH meter (Hanna, HI98103, USA), while the higher heating value (HHV) of the bio-oil was determined using a bomb calorimeter (CAL3KAP, South Africa). The biomass analysis employed gas chromatography mass spectrometry (GC-MS/QP2010, Shimadzu, Japan), while Helium was used as the carrier gas in a 5 MS capillary column that was 0.25 mm in diameter, 0.25 micrometer thick in film and 30 m long. A 1 µL sample volume was injected using the AOC-20i auto injector, the injector detector temperature was set to 280 °C. The following temperature program was used to determine the GC oven's temperature after being held at 80 °C for 2 minutes, the temperature was ramped up to 300 °C at the rate of 10 °C/minutes for 6 minutes to detect chemical compounds present in the bio-oil. These analyses revealed significant variations in the bio-oil compositions which could influence their stability for different uses, such as fuel protection or as feedstock for chemicals synthesis.

RESULTS AND DISCUSSION

Influence of the catalyst on the yield of bioproducts

Impact of microwave power level and catalysts (HZSM-5 and TiO_2)

Catalyst type is especially relevant as comparison between HZSM-5 and TiO_2. The data presented in Figure 3 show that at a power level of 500 W for 20 min and a particle size of 0.5 mm, both catalysts HZSM-5 and TiO_2 showed similar distributions of biochar and bio-oil. Although TiO_2 shows a slight increase in bio oil yield, the modest catalytic activity of TiO_2 likely facilitated the controlled thermal decomposition of biomass without causing significant secondary cracking reactions. When the microwave power was increased to 600 W, however, clear differences between the two catalysts became evident, the addition of HZSM-5 led to an evident increase in biogas yield along with a decrease in bio-oil yield (Ke et al., 2024), there are several reasons for this, in which the combination of strong acid sites and shape-selective properties of HZSM-5 promotes catalytic cracking and over cracking, also higher microwave powers will accelerate bond cleavage. Additionally, at higher microwave powers, vaporizable intermediates will be more quickly converted from a condensed phase to lighter, noncondensable gases, thus lowering bio-oil yields (Rueangsan et al., 2025).

It can be shown from Figure 3 that at a power level of 600 Watt and a particle size of 0.5 mm, TiO_2 exhibited a more balanced behavior; it achieved a much higher bio oil yield at higher microwave powers than HZSM-5, in which Bio-oil 32.42%, biogas 36.15%, and biochar is 31.43% when using TiO_2, while bio-oil decreases to 24.8% when using HZSM-5, but biochar and biogas increase to 34.3%,40.9%, respectively, and this is agreed with Wang et al (2018). The TiO_2 weak acidity and redox capabilities provide a favorable environment for partial deoxygenation and mild cracking while avoiding intense secondary reactions (Chanathaworn, 2022), therefore, TiO_2 will allow a larger proportion of intermediate vapors to be retained in their liquid phase before undergoing further degradation into permanent gases, while a reduction in the power level from 600 Watt to 500 Watt and the use of HZSM-5 as a catalyst showed an increase in the percentage yield of bio-oil from 24.8% to 25.7%. Likewise, the proportion of biochar rise from 34.3% to 41.1%. However, gas production

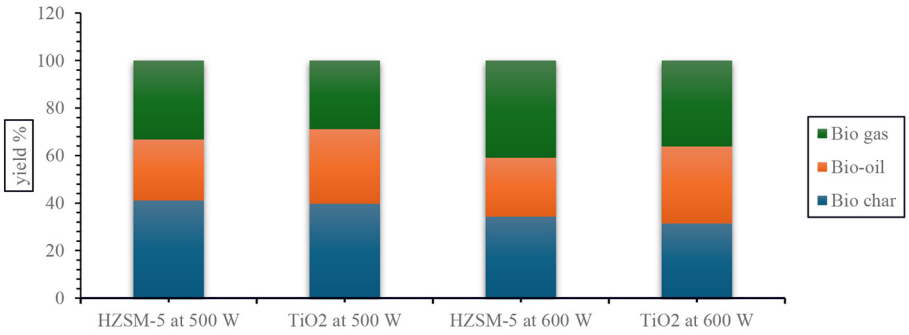

Figure 3. Impact of power level on bio-production

dropped from 40.9% to 33.2%, while, the addition of HZSM-5 at large power causes an increase in the temperature, which produces a decrease in the production of biochar and bio-oil and a substantial increase in the production of gaseous fuels (Wang et al., 2019).

The catalyst allows heat generated by the magnetron to pass to it by convection and subsequently transfers it to the biomass by conduction. This heat transfer mechanism would allow the biomass to better absorb microwave radiation. Due to this heat transfer, the reaction can be accelerated and reach completion sooner than it would without a catalyst. In addition, each type of catalyst will have an effect on what types of products are created (Abd and AL-Yaqoobi, 2025).

These findings align with those of Syed et al. (Syed et al., 2023) who reported that employing a catalyst reduces the energy needed to produce bio-oil and biochar, which in turn raises the final temperature (Kim et al., 2014).

Influence of reaction time

Figure 4 illustrates the impact of reaction time on bio-oil output, it can be shown a steady increase for both HZSM-5 and TiO_2 catalysts. At short reaction times (5 minutes), TiO_2 produces significantly more bio-oil than HZSM-5, with 24.4% compared to 15.4%. Its moderate catalytic activity effectively manages the initial stages of pyrolysis, this indicates that TiO_2 encourages efficient early reactions such as depolymerization and gentle cracking, leading to the quick formation of volatile condensables (Sharma et al., 2015).

As the reaction time increases to 10 and 15 minutes, the bio-oil yield continues to rise for both catalysts 20.2% and 23.7% for HZSM-5, 27.7% and 30.7% for TiO_2 respectively. However, the increase rate is still faster with TiO_2, showing a more controlled reaction and fewer intense secondary reactions. Meanwhile, the rise with HZSM-5 is slower because of early catalytic secondary reactions driven by its strong acidity (Ahmad et al., 2026) At longer reaction times (20 minutes), the bio-oil yield stabilizes, especially with HZSM-5, indicating an increased contribution from secondary processes, such as cracking, hypercracking, and polymerization, which constrain the increase in bio-oil yield (Nishu et al., 2020). The strong acidity of HZSM-5 speeds up these reactions, causing a partial conversion of condensable vapors into lighter gases or heavier feedstocks for carbon formation, thereby restricting bio-oil accumulation (Lu et al., 2014).

In contrast, TiO_2 maintains a higher bio-oil yield at extended reaction times, reaching 32.42% at 20 minutes, indicating that its moderate catalytic activity inhibits excessive secondary decomposition (Lu et al., 2014). The reduced tendency for excessive cracking allows for the condensation of a larger proportion of intermediate products into a liquid state. This behavior demonstrates how titanium dioxide (TiO_2) can expand the operational range for bio-oil production during prolonged reaction conditions (Mahmoud Fodah et al., 2021; Reza et al., 2023)

Effect of the catalyst weight percentage

The pyrolysis of biomass raw materials was carried out using microwave with different weight percentages of HZSM-5 solid acid catalyst, as shown in Figure 5a. At a power level of 500 W, varying the amount of catalyst used (from 20% to 50% by weight) significantly influenced the types of products formed and their yield percentages. The bio-oil yield decreased as the catalyst percentage increased from 26.2% to 23.65%, while the biogas yield increased from 28.7% to 46.1%. Residue yield decreased from 45.1% to 30.25%

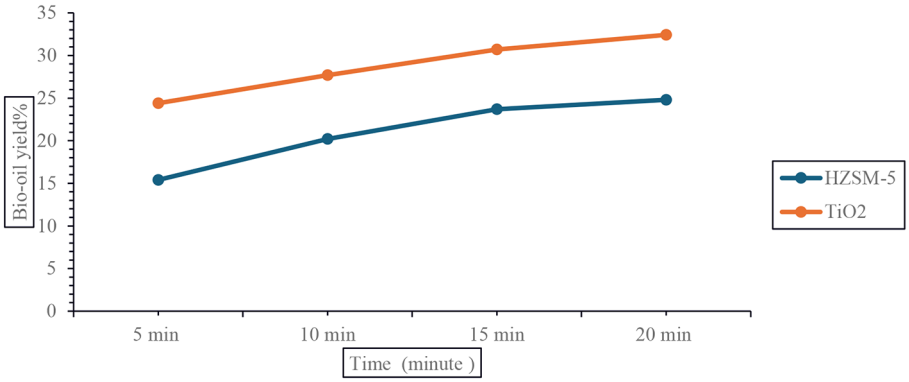

Figure 4. Effect of time on bio-oil yield %

as catalyst amount increased. These changes mainly result from initial microwave-assisted pyrolysis reactions, which involve over-breaking down of biomass into light, non-condensable gases, reducing condensable vapor production; (Cai et al., 2020), while coking effects from higher catalyst levels, causing blockage of active acid sites by carbon buildup; and faster reaction rates with more catalyst, which restricts mass transfer and hampers effective interaction between vapors and catalytic sites (Hu et al., 2020). Previous research has shown a consistent decline in bio-oil yield with increasing HZSM-5 catalyst loading, highlighting its effectiveness in promoting deoxygenation and aromatization reactions. (Ahmad et al., 2026; Rahman et al., 2018)

On the other hand, the catalytic activity of titanium dioxide was examined at the same percentages (20%, 30%, and 50%). Each experiment was conducted under identical conditions: 600 W of power was used. Figure 5b illustrates the effect of the catalyst dose on product yield. Bio-oil yield increased steadily with increasing catalyst dose. For example, bio-oil yield reached 29.3% with 20% catalyst. It was also found that bio-oil yield increased to 32.42% with 30% catalyst. Finally, bio-oil yield decreased to 32.37% with 50% of this dose (Chanathaworn, 2022).

Effect of catalyst on bio-oil properties

Physical analysis

Catalysts used to perform microwave pyrolysis will increase the quality of bio-oil that is generated by reducing oxygen levels within it through a deoxygenation process. As such, these catalysts contribute positively to the bio-oil's physical properties—including its thermal stability, heating values, and overall energy density. Therefore, it is crucial to measure the density (Fan et al., 2020). Bio-oils comprise carboxylic acids, including acetic, formic, and oleic acids, indicating high acidity; these acids can corrode or cause issues for use as fuels and chemical feedstocks. The catalytic process may impede the devolatilization of hemicellulose in the feedstock, thereby reducing the amount of organic volatile compounds in the liquid product and reducing the amount of acid formed (Abd and AL-Yaqoobi, 2025). A catalyst can enhance the production of lighter aromatic compounds and reduce the viscosity of the resulting bio-oil; both effects may result from the acceleration of pyrolysis reactions through the adsorption of reactants onto the catalyst, thus producing a larger portion of lighter compounds in the bio-oil, as indicated by Fodah et al. (Mahmoud Fodah et al., 2021) Furthermore, a catalyst may also lower the oxygen element content of the bio-oil, thereby raising the calorific value (Lin et al., 2018).

The influence of catalysts on the main properties of bio-oil produced through the microwave pyrolysis process is presented in Table 1. Based upon the explanation of the oil's attributes, it is evident that using HZSM-5 provides the least viscous and dense oils with the greatest pH values and calorific values. This is due to the production of light, volatile, low-oxygen, low-acid-content products from zeolites. Pyrolysis oils produced from *Dodonaea viscosa* using microwave-assisted pyrolysis showed that their physical and chemical properties are significantly influenced by the type of catalyst used.

The calorific value of the improved oils is 25.4 MJ/kg using the HZSM-5 catalyst and to 21 MJ/kg using the TiO_2 catalyst. Simultaneously,

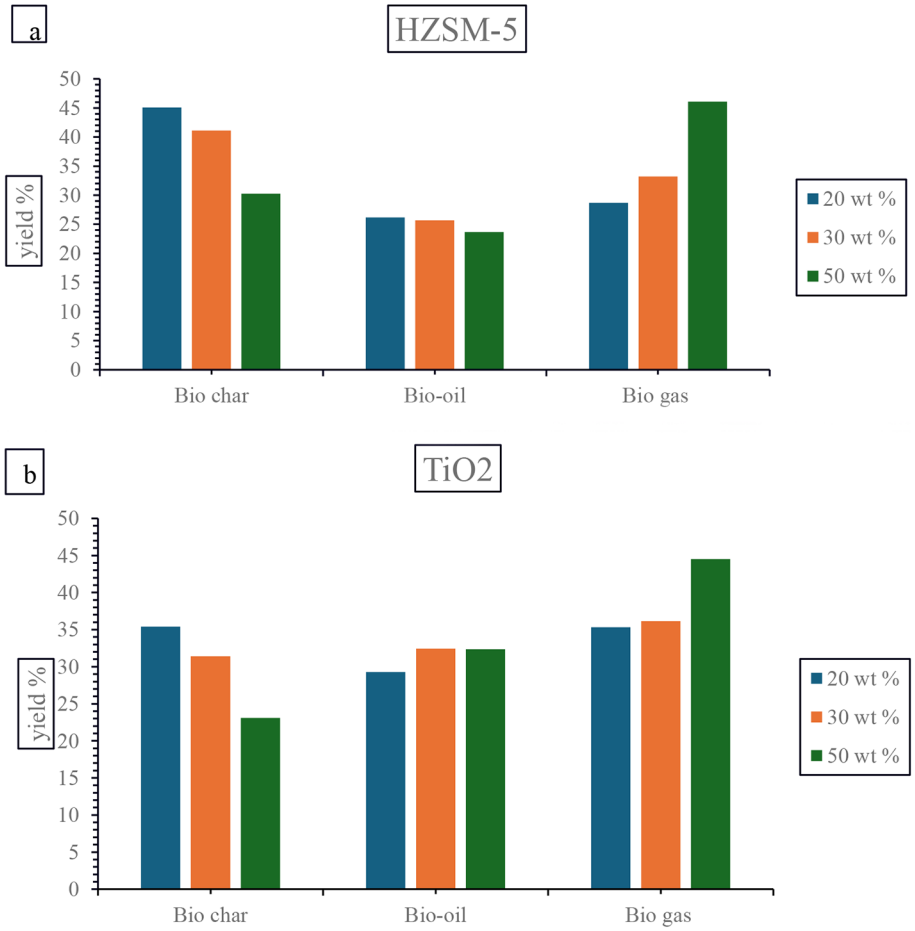

Figure 5. Effect of catalytic pyrolysis on product yield. a) HZSM-5, b) TiO_2

the pH of the improved oils reached 4.8 using HZSM-5 and 4.2 using TiO_2. Additionally, the breakdown of higher-molecular-weight molecules into lower-molecular-weight compounds reduced the density and viscosity of the enhanced oils. Improving the shortcomings of the pyrolysis oil, including its lower calorific value, high acidity, high viscosity, and low density, contributed to its improved combustion efficiency (Abdulkhani et al., 2023). The enhanced physical and chemical properties of bio-oil extracted with the HZSM-5 catalyst are attributed to the presence of robust acidic sites on its surface, facilitating the enhancement of deoxygenation and aromatization reactions in the thermal decomposition products. In contrast to the HZSM-5 catalyst, titanium dioxide (TiO_2) had a greater effect as a catalyst (Huynh et al., 2021).

Chemical analysis of bio-oil

The results from the GC–MS indicate clearly there are major differences in the chemical compositions of the bio-oils produced with HZSM-5 and TiO_2 catalysts; this demonstrates how much catalyst characteristics can affect the pathways for the conversion of biomass-derived oils and the selectivity of products formed.

HZSM-5 produces bio-oil containing large amounts of aromatic compounds while reducing significantly the concentration of acidic compounds and ketone compounds. This is largely due to both the high strength of the Brønsted acid sites and the pore structure of HZSM-5 (Ahmad et al., 2026). Both of these features enable deoxygenation reactions (which include decarboxylation, decarbonylation, and dehydration), as well as the aromatization of reaction intermediates (Garba et al., 2018). Figure 6 shows the increased amount of aromatic compounds is indicative of an efficient upgrading process that leads to improved fuel quality and higher energy density. Upon the introduction of HZSM-5, aromatic compound concentrations (i.e., benzene, toluene, xylene, and polyaromatics) have been shown 21.3%, while phenolic compounds is 21.57%.

Table 1. Comparison of physical properties of pyrolysis oil with catalyst

Catalyst	pH	Viscosity (cSt)	Density (Kg/m³)	HHV (MJ/Kg)
HZSM-5	4.8	4.38	990	25.4
TiO_2	4.2	5.57	1091	21

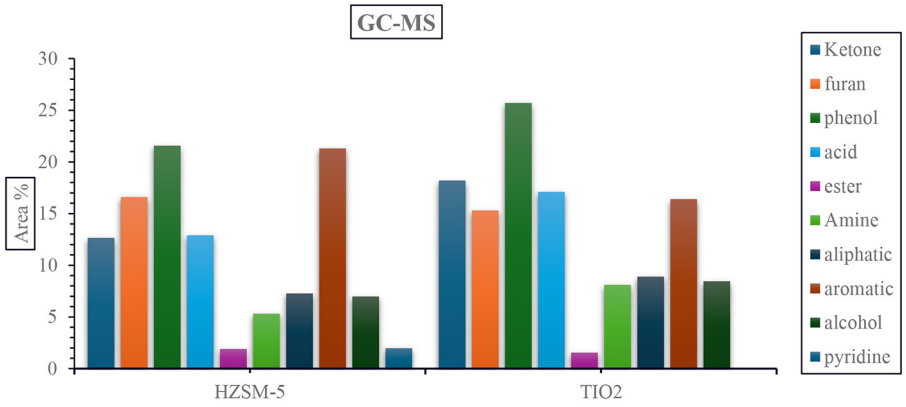

Figure 6. Difference between the chemical compositions of bio-oil

Depending upon the chemical composition, bio-oil may have a wide range of possible end uses. For example, if the bio-oil is primarily made up of phenolic and aromatic compounds, then the bio-oil can be treated as an alternative chemical feedstock. Conversely, if the bio-oil has a significant amount of hydrocarbon content, then it can be used as a fuel or further processed into a biofuel (Abd and AL-Yaqoobi, 2025). The presence of these aromatic compounds is of interest for enhancing the properties of Catalytic (Hernando et al., 2017).

Conversely, TiO_2 exhibits a different type of catalytic action. The bio-oil contains a higher concentration of oxygen-containing compounds, particularly phenolic compounds and ketones compounds. It appears that TiO_2 facilitates partial deoxygenation rather than extensive catalytic cracking. The relatively low acidity of TiO_2 along with the presence of redox-active sites allows for less severe reaction conditions, thereby preventing oxygenated intermediate compounds from being converted extensively to hydrocarbons (Zhang et al., 2018).

Additionally, it was shown that the distribution of aliphatic and nitrogen-containing compounds (including amines and pyridine-based derivatives) shows that TiO_2 preserves a wider range of chemical structures in comparison to HZSM-5, which preferentially directs reaction intermediates towards aromatic hydrocarbon formation. This preference for aromatic formation over other types of compounds is likely due to the pore confinement effects exhibited by the micropore structure of HZSM-5, where cyclization and aromatization occur preferentially. (Arabiourrutia et al., 2024). HZSM-5 performs excellently in converting biomass-derived oil through deoxygenation and boosting hydrocarbon production, while TiO_2 retains a larger portion of the intermediate compounds because of its lower catalytic efficiency (Zhang et al., 2019). Besides highlighting the importance of choosing the right catalyst to maximize yields, the results also show that catalyst selection significantly influences not just the product yield but also the chemical quality of the final output bio-oil.

CONCLUSIONS

This study examined the impact of two catalysts, specifically TiO_2 and HZSM-5, on the microwave-assisted pyrolysis of *Dodonaea viscosa* biomass for bio-oil production. The impact of critical operational parameters, including microwave power level, reaction time, and catalyst loading, were systematically evaluated in terms of product yield and quality. The results demonstrated that microwave power and reaction time are the most critical factors affecting product formation. Prolonged reaction times (exceeding

20 minutes) were found to promote secondary reactions, including cracking and condensation, that may diminish the yield of desirable liquid products. Furthermore, catalyst loading significantly influenced the distribution between liquid and solid products. An optimal catalyst loading of 30 wt% was identified, providing the best compromise between conversion efficiency and product selectivity.

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