PRODUCTION OF VALUE-ADDED BIOCHAR AND BIO-OIL FROM MICROWAVE CO2 PYROLYSIS OF MEDIUM-DENSITY FIBERBOARD WASTE

SHIN YING FOONG¹, FELICIA LOW¹, PETER NAI YUH YEK^{2*}, YAN YANG¹, LINGBO MENGⁱ, ROCK KEEY LIEW³, MEENAKSHI VERMA⁴, NATARAJAN RAJAMOHAN⁵AND SU SHIUNG LAM1,6*

1 Higher Institution Centre of Excellence, Institute of Tropical Aquaculture and Fisheries, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia. ²Centre for Research of Innovation and Sustainable Development, University of *Technology Sarawak, No.1, Jalan Universiti, 96000 Sibu, Sarawak, Malaysia. 3 NV WESTERN PLT, No. 208B, Second Floor, Macalister Road, Georgetown, 10400 Penang, Malaysia. 4 University Centre for Research and Development, Department of Chemistry, Chandigarh University, Gharuan, Mohali, 140413 Punjab, India. 5 Chemical Engineering Section, Faculty of Engineering, Sohar University, 311 Sohar, Oman. 6 Faculty of Engineering, Sohar University, PO Box: 44, PC: 311 Sohar, Oman.*

**Corresponding authors: lam@umt.edu.my, peter.yek@uts.edu.my http://doi.org/10.46754/jssm.2024.06.006 Submitted final draft: 4 February 2024 Accepted: 11 March 2024 Published: 15 June 2024*

Abstract: This study investigates the feasibility of using microwave CO₂ pyrolysis to dispose of medium-density fibreboard (MDF) waste, a significant by-product of the furniture manufacturing industry. The primary goal is to transform MDF waste into valueadded biochar and bio-oil, exploring an alternative to conventional pyrolysis methods that typically use nitrogen (N_2) as a purge gas. The research emphasises the potential benefits of $CO₂$ utilisation in pyrolysis, both to enhance the quality of biochar and bio-oil and as a strategy for greenhouse gas (GHG) mitigation. This research reveals that conducting microwave pyrolysis in a CO₂ atmosphere increases biochar (up to 44.8%) and bio-oil (up to 21.2%) yields over N_2 pyrolysis. CO_2 hinders volatile thermal cracking and aids in gas defragmentation and repolymerisation, with biochar yield rising with time and biooil peaking at an optimal duration. This study underscores microwave CO_2 pyrolysis as a promising alternative for converting biomass waste into valuable products, potentially contributing to fuel utilisation. It also highlights the environmental benefits of this method, particularly in terms of CO_2 utilisation, aligning with the broader objectives of sustainable waste management and renewable energy production. The findings provide a basis for future research in this area, focusing on optimising process parameters, exploring potential applications of bio-products, and assessing the economic and environmental viability of this innovative approach.

Keywords: Furniture board, waste recovery, waste furniture, recycle, medium-density fibreboard (MDF).

Introduction

The world population has been growing continuously for the last 50 years, resulting in a huge increase in demand for primary energy (Milano *et al*., 2016). As a country that relies mainly on fossil fuels for its development, Malaysia could be affected by the depletion of fossil fuels (Ahmad *et al*., 2011). Besides that, the world's energy consumption is mainly supplied by fossil fuel, a non-renewable source that may lead to an energy crisis (Lo *et al.*, 2017). Moreover, the extensive consumption of fossil fuels has led to environmental issues, including the emission of large quantities of carbon dioxide, a greenhouse gas contributing

to global warming. Consequently, the world is grappling with two major problems: An energy crisis and environmental degradation. This situation urgently calls for alternatives to produce bioenergy and decrease dependency on fossil fuels.

Besides the aforementioned problem, waste production is another global issue that needs to be resolved. A huge quantity of lignocellulosic waste is produced globally from numerous sources, including the furniture industries, contributing to environmental problems (Małaszkiewicz *et al.*, 2018). Medium-density fibreboard (MDF) is extensively utilised in the construction and furniture sectors, with global production rates reaching approximately 22 m^3 and continuing to rise (Karade, 2010). Due to its comparatively shorter lifespan than other construction materials such as bricks, wood and concrete, a significant volume of MDF becomes waste after its use period. This volume is further augmented by the inclusion of cut-off wastes produced during manufacturing and fabricating. These wastes are commonly disposed of through burning or landfilling, leading to several environmental issues, including air pollution, global warming, and land occupation. Moreover, the lignocellulosic wastes in landfills undergo biodegradation and emit methane. Methane is a greenhouse gas with a warming effect 72 times greater than CO_2 (Lelieveld *et al*., 1992). Recycling of MDF waste is challenging as the size of the particles of the wood is dust-like, while combustion of MDF waste causes the formation of toxic gases due to the resins such as melamine-formaldehyde and urea-formaldehyde that are used as glue and coating (Girods *et al.*, 2009). In many countries, the combustion of MDF is forbidden because free formaldehyde content poses carcinogenic properties (Chen *et al*., 2016). Hence, pyrolysis is one of the promising approaches for waste recovery.

Pyrolysis, a high-temperature, inertenvironment thermochemical process, decomposes feedstock to produce biochar, gases, and bio-oil. It efficiently valorises biomass waste, notably complex materials like medium-density fibreboard (MDF) (Aslan *et al.*, 2018). Microwave-assisted pyrolysis (MAP) is a promising approach compared to conventional pyrolysis (CP) due to its faster reaction rates and higher energy efficiency (Li *et al.*, 2016). Microwaves induce molecular motion to generate heat due to the friction between particles by moving ionic species, rotating dipolar species, or both (Haque, 1999). A notable feature of microwave heating is the creation of hotspots, resulting in localised temperatures within the feedstock that are higher than those recorded in the bulk material (Huang *et al.*, 2016). Overall, microwave pyrolysis has a high potential for energy recovery. However, MAP could possess limitations such as the need for nitrogen as flow gas to create an inert environment which increases the operation cost of MAP. Alternatively, carbon dioxide (CO_2) can be used as a flow gas.

The use of $CO₂$ as an activation agent in microwave pyrolysis for producing bio-oil and biochar, particularly from MDF, presents a fascinating area of research within the field of biomass energy conversion. This process capitalises on the inherent advantages of microwave heating, such as selective heating and rapid temperature rise, to enhance the pyrolysis process (Liew *et al.*, 2018). Conventional pyrolysis suffers from slow, uneven heat transfer, causing variable product quality. Microwave heating offers uniform distribution and targets specific biomass components for controlled pyrolysis. Adding $CO₂$, a mild oxidant, further impacts the process, reacting with biomass to affect the yield and quality of bio-oil and biochar, adding complexity and potential benefits to the procedure (Lin *et al.,* 2015; Chen *et al.,* 2022).

Microwave $CO₂$ pyrolysis applied to MDF, a prevalent wood industry waste comprising wood fibres, resin, and wax, offers significant advantages. MDF's complex composition poses challenges in biochar and bio-oil production. Microwave heating efficiently breaks down these intricate bonds, outperforming traditional methods. Additionally, incorporating $CO₂$, a notable greenhouse gas, in pyrolysis aligns with carbon capture and sequestration strategies, enhancing environmental benefits (Lin & Chen, 2015; Chen & Lin, 2016). This technique not only optimises the pyrolysis process but also aids in reducing $CO₂$ emissions. It epitomises the principles of biomass utilisation and waste-towealth initiatives, emphasising environmental sustainability. Recently, studies have been conducted on pyrolysis that employed $CO₂$ instead of N_2 as carrier gas. For instance, Zhang *et al.* (2011) reported that pyrolytic oil produced under a $CO₂$ atmosphere is rich in acetic acid and suppresses the formation of phenol. Biswas *et al*. (2018) reported an increase in pyrolytic

product yield under a CO_2 atmosphere compared to an N_2 atmosphere, and the bio-oil produced contains high amounts of oxygenate and low aliphatic compounds. The biochar produced under the $CO₂$ atmosphere was also proven to have a well-developed pore structure and high porosity (Jung *et al.,* 2019; Luo *et al.*, 2019).

However, most pyrolysis studies under a CO2 atmosphere were performed with CP. To the best of the author's knowledge, a limited number of studies were conducted on microwave $CO₂$ pyrolysis and its potential impact on the pyrolysis reaction. This study uses MDF waste as a feedstock for microwave CO_2 pyrolysis to produce value-added biochar and bio-oil. CO₂ serves as a reaction medium, enhancing thermal efficiency while mitigating environmental issues such as global warming resulting from high levels of $CO₂$ in the atmosphere (Kwon *et al*., 2013). This study aimed to narrow the existing knowledge gap regarding the impact of various pyrolysis atmospheres on the yield of pyrolytic products. For researchers, it provides an opportunity to uncover differences or advantages associated with employing $CO₂$ as a carrier gas or reactive medium in microwave pyrolysis, which has not been thoroughly examined in prior studies. Consequently, this exploration may lead to the development of a novel technique in microwave pyrolysis.

Materials and Methods

Materials Preparation

Medium-density fibreboard (MDF) was obtained from a furniture manufacturer in Zhengzhou, China. The MDF was crushed into smaller fragments (2-3 cm) and oven-dried at 80°C for 24 h to eliminate the retained moisture. The samples were then stored in a sealed bag before the pyrolysis process.

Experimental Setup of Microwave-assisted Pyrolysis

Microwave pyrolysis of MDF wastes was conducted via a modified microwave oven, as reported in previous research (Wan Mahari *et al*., 2018). The system was made up of four components: (i) A magnetron that emits

microwave radiation, (ii) a quartz reactor (150 x 100 x 100 mm), (iii) a type-K thermocouple connected to a temperature controller, and (iv) a condensation system for bio-oil collection. A 50 g MDF sample was weighed and placed in the quartz reactor. A ceramic fibre blanket insulated the pyrolysis reactor to minimise heat loss during the heating and pyrolysis stages. A type-K stainless-steel thermocouple, connected to a temperature controller, was utilised to monitor temperature variations throughout the pyrolysis of MDF wastes. The pyrolysis experiments were conducted under N_2 and CO_2 atmosphere with three different reaction times (20 min, 30 min, and 40 min) under the same power of 800 W. N₂ and CO_2 gases at a flow rate of 0.5 L/min were purged into the pyrolysis reactor along pyrolysis process to maintain an inert environment. The char residue yield was determined by calculating the weight difference of the pyrolysis reactor before and after the pyrolysis process, whereas the weight of the liquid oil was obtained by measuring the contents of the collection vessel.

Thermogravimetric Analysis

Mettler Toledo thermogravimetric analyser was used to conduct thermogravimetric analysis of MDF waste. The MDF wastes were pyrolysed in N₂ condition at 10°C/min heating and 25 mL/ min gas flow rates from room temperature to 900°C. The chemical reactions and proximate content were determined based on the TGA curves obtained. The TGA curves obtained were also used to determine the proximate content and minimum pyrolysis temperature for optimal recovery of biochar of MDF wastes.

Calculation of Microwave Pyrolysis Yield

The yield of pyrolytic products from a biomass feedstock during pyrolysis was calculated based on the mass of each product obtained relative to the initial mass of the dry biomass feedstock. The biochar process begins by recording the weight of the dry biomass feedstock. This mass serves as the denominator in yield calculation. After

undergoing microwave pyrolysis, the residual solid, which is the biochar, is allowed to cool to room temperature to prevent any changes in mass due to oxidation or further reactions. The biochar is then weighed, and its mass compared to the initial biomass mass gives biochar yield in percentage by weight. Bio-oil measurement involves condensing the volatile compounds released during pyrolysis. These vapours pass through a cooling system, usually a series of condensers, which then liquefy into bio-oil. The bio-oil is a complex mixture of organic liquids and requires careful separation from water and other non-condensable gases. Equation 1 shows syngas yield (Y_{syngas}) calculation in weight per cent (wt%). Y_{biochar} is the yield of biochar, and $Y_{\text{bio-oil}}$ is the yield of bio-oil, all calculated as a percentage of the initial dry biomass mass. The syngas yield is calculated assuming no other significant by-products or losses exist.

$$
Y_{\text{syngas}}\left(\text{wt}\%\right) = 100\% - \left(Y_{\text{biochar}} + Y_{\text{bio-oil}}\right) \quad (1)
$$

Results and Discussion

Thermogravimetric Analysis of MDF

Figure 1 presents the thermal degradation behaviour of MDF waste. The TGA curve was plotted in blue while the DTG curve was plotted in orange. Based on Figure 1, the thermal decomposition of MDF waste can be distinguished into three stages: The evaporation of moisture, de-volatilisation, and carbonisation (Liew *et al.*, 2018). In the first stage, some weight loss occurred below 100°C. This was likely due to the loss of retained water within the MDF waste. The weight decreases slightly at temperatures between 110°C to 250°C could be the decomposition of small organic molecules $(e.g., CH₄)$ in MDF waste.

From the TG-DTG curve, there was a significant weight loss between the temperature from 265°C to 380°C, attributed to the breakdown of cellulose and hemicellulose in the MDF waste (Jiang *et al*., 2014). It was also known as the devolatilisation process. This temperature zone accelerated the depolymerisation process, such as the glycosidic bond in cellulose, which broke easily at this stage (Jiang *et al.*, 2014). Besides that, other carbonaceous matters such as CO and $CO₂$ start to form at this stage. According to Tzvetkov *et al.* (2016), the lignocellulosic component has different thermal stability and decomposes at varying temperature ranges. The hemicellulose decomposes at 180°C to 285°C, cellulose decomposes at the range of 285°C to 365°C, while lignin decomposes at 365°C to 500°C as it is the most thermally stable lignocellulosic component.

Figure 1: TG (blue) and DTG (black) curves of MDF waste

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After that, there was another slight decrease in weight loss between 400 to 800°C, attributed to the release of lower molecular weight carbonbased compounds. These emissions often include carbon oxides $(CO_x$ gases), hydrocarbons in the form of tar (C_xH_y) , and incondensable gases like hydrogen (H_2) . These substances typically originate from the non-volatile residue after the devolatilisation phase, undergoing a charring process. This steady and small weight loss could be considered as the complete evaporation of the non-carbonaceous materials from the biochar and the leftover solid was the carbon-dense product (biochar). Given the complexity of biomass components, accurately characterising the thermal decomposition process poses a challenge.

Proximate Content of MDF

Table 1 presents the proximate content of MDF waste. By analysing the data presented in the table, it is evident that MDF waste exhibits significant levels of volatile matter (51 wt\%) , alongside considerable amounts of fixed carbon (21 wt\%) and ash (24 wt\%) , with a relatively low moisture percentage (4 wt\%) . The prevalence of volatile matter at 51% by weight in MDF waste, while not optimal for producing carbon-rich solid char, is favourable for the generation of bio-oil or biogas. These can be subsequently refined into fuels or energy. This study, however, focuses solely on producing solid char and liquid bio-oil. The carbonisation process leads to the elimination or evolution of volatile compounds from MDF waste, which plays a key role in developing porosity on biochar surfaces. The formation of these volatile compounds is attributed to the breakdown of

the lignocellulosic elements, undergoing a series of chemical transformations that include dehydration, breaking of chemical bonds, and reorganisation of molecular structures (Yang *et al.,* 2018). The high content of fixed carbon (i.e., non-volatile composition) in MDF also indicated that MDF can be converted into biochar through pyrolysis.

A relatively high amount of ash (up to 24 wt%) was detected in the MDF waste. The ash composition in biomass includes potentially beneficial inorganic elements like Na, K, Al, Fe and Mn. These elements can function as inorganic catalysts during the pyrolysis process. Their catalytic action can improve the cracking of biomass, facilitating the release of a greater amount of volatile matter. This enhancement in the pyrolysis process due to the presence of these elements can significantly influence the efficiency and outcome of biomass conversion. Overall, MDF is suitable as feedstock for pyrolysis to produce biochar and bio-oil.

Table 1: Proximate contents (wt%) of MDF waste

Proximate Content (wt%)				
Volatile matter	51			
Fixed carbon	21			
Ash	24			
Moisture				

a Calculated by difference (Fixed carbon = 100 wt% – volatile matter – ash – moisture)

Comparison of Microwave Pyrolysis Yield

The results presented in Table 2 for microwave pyrolysis experiments aimed at producing biochar, bio-oil, and syngas from biomass offer insightful information, particularly when

Table 2: Yields of biochar, bio-oil, and syngas from microwave pyrolysis under CO_2 and N_2 atmospheres at different reaction times

Time (min)	CO.						
Products	20	30	40	20	30	40	
Biochar $(wt\%)$	44.8	44.0	43.5	43.5	43.7	42.7	
Bio-oil $(wt\%)$	19.8	21.2	20.2	16.8	17.2	15.9	
Syngas ($wt\%$)	35.4	34.8	36.3	39.7	39.1	41.4	

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considering the impact of varying atmospheres $(CO₂$ and $N₂$) and durations (20, 30, and 40 min) on the process. It is important to delve into the implications of these variations, especially the observation that longer durations lead to higher temperatures and how this influences the yield and quality of the products.

Biochar Yield

The biochar yield is higher in a $CO₂$ atmosphere than N_2 during microwave pyrolysis due to different reaction mechanisms and the influence of reactive atmospheres. In a CO_2 atmosphere, the reactions may involve carbon dioxide participating actively in the pyrolysis process. $CO₂$ is known to react with carbon to form carbon monoxide (CO) at high temperatures due to the Boudouard reaction (Kwon *et al.*, 2019). This endothermic reaction can absorb heat, reducing the reaction rate and potentially leading to a more controlled pyrolysis process. This control could facilitate the formation of a more stable char structure, hence increasing the yield. In contrast, an N_2 atmosphere is relatively inert and does not participate in the chemical reactions during pyrolysis. This inertness might lead to a more straightforward biomass degradation, where primary reactions dominate, and biochar is subjected to further conversion into bio-oil and syngas as the process progresses, especially at extended reaction times. The differences in biochar yield between $CO₂$ and $N₂$ atmospheres highlight the role of gas-solid interactions in pyrolysis. $CO₂$ seems to engage in pyrolysis chemistry, altering the trajectory of decomposition and enhancing char formation or preservation.

Bio-oil Yield

The final yield of bio-oil produced under $CO₂$ atmosphere at 800 W microwave power is shown in Figure 2. As shown in Figure 2, biooil obtained at 30 min has the highest yield (21.2 wt\%) , while bio-oil obtained at 20 min has the lowest yield (19.8 wt\%) . It is noted that a lower yield of bio-oil was obtained at a shorter reaction time. The long reaction time increased the bio-oil yield because the volatiles retained in the pyrolysis system were effectively condensed, resulting in less gas formation. The highest bio-oil yield was achieved at 30 min (21.2 wt%), whereby further extended reaction time did not enhance bio-oil production. *Yan et al.* (1999) reported a similar phenomenon in that an insignificant increase in bio-oil yields for longer reaction times was noticed. Therefore, the optimal pyrolysis time was 30 min for maximum bio-oil yield.

The observed increase in bio-oil yield with time in a $CO₂$ atmosphere and its decrease in an N_2 atmosphere during microwave pyrolysis is a fascinating aspect that merits deeper analysis. Bio-oil is a complex mixture of organic compounds, and a series of primary and secondary reactions influence its formation during pyrolysis. In a $CO₂$ atmosphere, the increased bio-oil yield due to the CO_2 might be participating in the reactions as a mild oxidant, which can lead to partial oxidation of the biomass. This partial oxidation could facilitate biomass breakdown into liquid components rather than gas, thus increasing the bio-oil yield. Another aspect could be the potential role of $CO₂$ in stabilising reactive intermediates that lead to bio-oil formation, preventing their further breakdown into non-condensable gases. $CO₂$ could also act as a blanket, reducing the peak temperatures achieved during pyrolysis, resulting in a different thermal degradation pathway favouring oil formation. Moreover, $CO₂$ might reduce secondary reactions that typically crack bio-oil into gases.

The bio-oil yield obtained from microwave $CO₂$ pyrolysis is higher than from the $N₂$ atmosphere. Wang *et al*. (2018) reported an increase in bio-oil yield obtained under $CO₂$ atmosphere and the bio-oil produced is rich in phenols. Wang *et al.* (2018) claimed that the increase in bio-oil yield under the $CO₂$ atmosphere was attributed to the enhanced conversion of methylene and hydroxyl groups to produce aliphatic and phenol compounds. An increase in free radicals such as \bullet CH₃, and \cdot CH₂ is produced from pyrolysis under the $CO₂$ atmosphere, which eases the bio-oil fragments stabilisation, and thus increases the bio-oil yield (Wang *et al.*, 2018). According to Tokmakov *et al.* (1999), these free radicals favour recombining with aryl groups to form light aliphatic hydrocarbons, thus enhancing bio-oil yield. According to Kwon *et al.* (2019), $CO₂$ enhanced the pyrolysis reaction by shifting the carbon distribution from pyrolytic oil. This is attributed to the reaction mechanistic roles of $CO₂$, which enhance the thermal cracking of volatile matters and the reaction between $CO₂$ and volatile matters. Overall, microwave pyrolysis under a CO₂ atmosphere can promote the decomposition of MDF to produce a higher bio-oil yield, and the optimum reaction time is 30 min for maximum bio-oil yield.

 $N₂$ being inert, does not actively participate in the pyrolysis reactions, which means that the primary pyrolysis reactions dominate. With time, these primary reactions can lead to secondary reactions where bio-oil components are further broken down into gases, as evidenced by increased syngas yield with longer reaction times. Secondary reactions in pyrolysis typically involve cracking volatile organic compounds into smaller molecules. Without the reactive influence of $CO₂$, these reactions may proceed uninhibited in an N_2 atmosphere, leading to a higher production of syngas and a corresponding decrease in bio-oil yield. This suggests that in N_2 , the thermal degradation of biomass is more complete, leading to a greater degree of depolymerisation and gasification, especially at higher residence times.

Syngas Yield

The increasing yield of syngas with time in both CO_2 and N_2 atmospheres during microwave pyrolysis indicates the progressive deconstruction of biomass. Syngas, primarily composed of hydrogen and carbon monoxide, is a central intermediate in the conversion of biomass to energy and chemicals, and its production is a key indicator of the efficiency of the gasification process. In the $CO₂$ atmosphere, the more pronounced increase in syngas yield

could be attributed to the Boudouard reaction, where CO_2 reacts with carbon to form CO, which is likely occurring at the high temperatures associated with pyrolysis (Kwon *et al.,* 2019). This reaction not only contributes directly to the syngas composition by increasing the carbon monoxide concentration but also implies that the presence of $CO₂$ can facilitate the gasification of biochar. Consequently, as the pyrolysis time increases, more biochar is converted into syngas components, enhancing the yield.

The increase in syngas yield with time in an N_2 atmosphere, while less pronounced than in $CO₂$, still supports the notion of continued biomass degradation. In an inert N_2 atmosphere, the biomass components break down primarily through pyrolytic decomposition, releasing volatile products and then reforming or cracking into syngas components. Over time, this process gradually increases syngas yield as more biomass is converted into gaseous products. The extended reaction times in both atmospheres likely contribute to the thermal cracking of heavier organic compounds and reforming these volatiles into syngas components.

Challenges and Future Perspectives

The advancement of microwave CO_2 pyrolysis, particularly for processing materials like medium-density fibreboard (MDF), heralds a new era in biomass waste management and renewable energy production. Yet, this field faces several critical challenges and areas for future development. Scaling up from laboratoryscale to industrial-scale operations in microwave CO₂ pyrolysis introduces complex issues. These include overcoming heat transfer limitations, achieving uniform microwave distribution, and maintaining an inert or CO_2 -rich atmosphere across larger processing volumes. Ensuring the biochar and bio-oil maintain consistent quality and yield when produced on a larger scale is complex and demanding.

Assessing the economic feasibility of microwave $CO₂$ pyrolysis is equally crucial. Although promising in enhanced bio-oil and biochar yields, the costs involved in microwave

technology, CO_2 acquisition, handling, and overall energy consumption need comprehensive evaluation against the process benefits. The economic viability of this method is pivotal for its integration and acceptance in the biomass waste management sector.

Evaluating the environmental impact of this pyrolysis technique is imperative. Utilising CO₂ in the process aligns with carbon capture and utilisation strategies, but a thorough analysis of the environmental footprint, including emissions during processing and a life cycle analysis of the bio-products, is essential. Such an assessment will confirm the technical viability and environmental sustainability of the process. The field should focus on optimising process parameters to maximise the yields and quality of biochar and bio-oil. Investigating innovative catalysts that can be used in conjunction with $CO₂$ in microwave pyrolysis might further boost the process's efficiency and selectivity. Investigating uses for biochar and bio-oil, such as soil improvement, carbon sequestration, or renewable fuels, is vital, offering insights and market prospects. Integrating microwave $CO₂$ pyrolysis into wider waste and renewable energy systems could lead to sustainable solutions, including using renewable energy for microwave power and employing biochar in environmental cleanup. These are promising avenues in the advancing field of biomass conversion, as well as supporting the United Nations Sustainable Development Goals 13 (climate action). The utilisation of $CO₂$ in microwave pyrolysis supports the core goal of SDG 13, which is to take urgent action to combat climate change and its impacts while supporting the circular economy by converting waste materials into value-added products.

Conclusions

This research marks a notable advancement in biomass waste management and renewable energy through microwave $CO₂$ pyrolysis, converting MDF waste into biochar and bio-oil. This method shows superiority over traditional pyrolysis, achieving higher yields and possibly

better environmental outcomes by utilising $CO₂$. It effectively produces more biochar and biooil than nitrogen-based pyrolysis, attributed to $CO₂$'s role in altering biomass degradation. The study identifies optimal conditions for maximum yields, guiding future experimentation, and industrial applications. However, scaling up, economic feasibility and environmental implications remain challenges. Future research should aim to refine process parameters, explore new catalysts, evaluate bio-product applications, and incorporate this technique into broader waste management and renewable energy frameworks. The potential of microwave $CO₂$ pyrolysis in contributing to sustainable waste management and energy production is significant, but it requires a concerted effort in research, development, and innovation to realise its full potential.

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Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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