

Research Article

Flash Pyrolysis Experiment on *Albizia odoratissima* Biomass under Different Operating Conditions: A Comparative Study on Bio-Oil, Biochar, and Noncondensable Gas Products

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This study deals with the flash pyrolysis of *Albizia odoratissima* biomass wastes at different temperature, sweep gas flow rate, and heating rate in a fluidized bed reactor. In the first phase of the experimental work, the effect of temperature (350–550°C) on product yield was analyzed, the second and third phases of the work were to analyze the effect of sweeping gas (N₂), flow rate (1.25–2.25 m³/hr), and heating rate (20–40°C/min). The experimental works were carried out to get maximum bio-oil production. The experimental results demonstrated that the maximum yield of bio-oil was obtained at a temperature of 450°C, N₂ flow rate of 1.75 m³/hr, and heating rate of 30°C/min. Temperature was found to be the crucial factor rather than sweep gas flow rate in the product distribution. Fourier transform infrared spectroscopy (FT-IR), gas chromatography mass spectroscopy (GC-MS), and elemental analysis were done on the obtained bio-oil, biochar, and noncondensable gas products. The heating value of the bio-oil and biochar was identified as 18.15 and 23.47 MJ/kg, respectively. The chemical analysis of the bio-oil showed that the oil is a mixture of phenol and oxygenated elements. The gas analyses showed that hydrogen and carbon dioxide were dominant, followed by carbon monoxide and methane.

1. Introduction

Finding alternative and sustainable energy to replace fossil fuels has been prompted for the past three decades by rising fossil fuel prices and resource depletion. Biomass is a

renewable carbonaceous material that has received widespread attention. It is available in a variety of forms and is used for power and chemical production [1]. Currently, the availability of biomass accounts for about 10% of the total global energy production [2], and its annual availability are

estimated at 100 billion tonnes per year. According to the IEA, biomass can replace 27% of the global transportation energy needs by 2050 [3]. Biochemical and thermochemical are the two efficient approaches for the production of biofuels. Methane and alcohol are the two main products of biochemical conversion techniques, whereas pyrolysis, gasification, combustion, hydrothermal liquefaction, and carbonization are the different types of thermochemical conversion methods [4, 5]. Among the various thermochemical conversion techniques, pyrolysis is considered as a most attractive method [6].

Slow and fast pyrolysis is the two different types of pyrolysis. Slow pyrolysis is a very old technique that has been used for millennia for the production of char for firing purposes [7]. Fast or flash pyrolysis is the most efficient technique which produces energy-rich bio-oil rather than the production of char and gaseous fuel. The yields of pyrolysis are strongly influenced by the reaction conditions. In fast pyrolysis, biomass decomposes fastly to release most of condensable vapours with a lower yield of char and gaseous components. The condensable vapours can be cooled to get dark brown liquid oil called pyrolysis oil [8]. Wood chips or agricultural leftovers from lignocellulosic biomass are commonly used feedstocks for biomass pyrolysis. Several studies have focused on the pyrolysis characteristics of various materials and their product characterization at the laboratory and industrial scale, including their reaction kinetics and modeling. However, the yield of biofuel and its quality basically depends upon the quality of feedstock and various operating parameters [9, 10]. For the past three decades, many articles have been published related to the pyrolysis of various biomass materials by using different types of reactors. Zhang et al. [11] reviewed various physicochemical characteristics of pyrolysis oil. The study identified the problems that must be addressed to improve the reaction. Isahak et al. [12] revised their findings on bio-oil by focusing on biomass characterization and the design of the reactor. Ahmed et al. [13] studied pyrolysis and thermal stability analysis of *Acacia auriculiformis* biomass to produce biofuels. Khuenkao and Tippayawong [14] conducted pyrolysis experiments on corncobs, coconut shells, and bamboo residue using an ablative reactor. The study investigated the production of pyrolysis oil and char by changing inert gas flow rate (nitrogen), rotating frequency, and reaction temperature. The study showed a higher bio-oil yield of 72 wt% with the corncob under the N₂ flow rate of 5 L/min and 6 Hz rotation frequency compared to the coconut shell and bamboo residues. Next to the corncob bio-oil, the coconut shell yielded 50 wt% of bio-oil under varying flow rate and frequency.

Fluidized bed technology is a good platform for pyrolysis of wood-based biomass due to its simple design, higher efficiency, higher contact surface, and shorter residence time [15]. Fluidized bed reactors are particularly designed for maximum oil production [16]. Many studies previously yielded more bio-oil when the mallee wood [17], beech wood [18], pine [19], and bamboo [20] biomass was pyrolyzed using fluidized bed reactors. From these literatures, it can be

understood that the pyrolysis reactor is the significant one to control product yields. Pattiya [21] conducted pyrolysis experiment on cassava residues using a fluidized bed reactor and produced 65 wt% bio-oil. Kim et al. [22] pyrolyzed *Jatropha* wastes and produced maximum oil fractions of 48 wt%. Park et al. [23] conducted fast pyrolysis of carbonaceous materials by utilizing sawdust and empty fruit bunches. The study reported that 60 wt% of oil was obtained at 500°C. In this series, Griffin et al. [24] discussed the uses of Australian acacias for biofuel production, and Madhu et al. [25] concentrated on production of pyrolysis oil from the cotton shell and empty fruit bunches. Wood and wood bark pyrolysis of *Albizia amara* conducted by Sowmya Dhanalakshmi and Madhu [26] showed maximum production of bio-oil of 48.5 wt%. The produced bio-oil was acetic in nature and they suggested the liquid oil as a feedstock for industrial chemicals. From the above said literatures, the fluidized bed pyrolysis of biomass produced maximum oil products compared to other pyrolysis reactors.

Albizia odoratissima is a tree that belongs to Fabaceae family. It is the fastest growing tree native to India, China, Bangladesh, and Sri Lanka. The wood of the tree is dark brown in colour, typically striped, sturdy, and dense. The wood can be used for making furniture and agricultural equipment. It grows up to 35 metres height with a short trunk. The bark of the tree is identified in dark grey with horizontal lenticels. The wood generally has 12 wt% moisture content, and it is 20–40% stronger than teak [27]. The trees have been widely planted as shade trees in tea and coffee estates. The physicochemical analysis of the biofuel obtained from various biomass materials is an important study to find its suitability and applications. Sahoo et al. [28] carried out a deep characterization study on biofuels obtained from rice straw, wheat straw, and sugarcane bagasse. The oil products obtained from these biomasses were acidic, with an overall calorific value of 10 MJ/kg. The authors suggested using this oil as a fuel due to its lower ash content and lower viscosity compared to commercial fuel. The SEM and CHNS analysis of the biochar showed it to be used as a fertilizer for soil to restore its nutrients. According to Charusiri and Vitidsant [29], the bio-oil produced from sugarcane leaves, cannot be utilized as a fuel for direct burning due to lower calorific value. But the cocatalytic pyrolysis process was recommended by the authors to produce potential biofuel and chemicals. *Azadirachta indica* was transformed into noncondensable gases using pyrolysis by Sowmya Dhanalakshmi and Madhu et al. [30]. The obtained gas was reported to have a higher amount of methane and carbon monoxide.

Accordingly to these findings, the authors of this paper decided to conduct pyrolysis experiments on *Albizia odoratissima* as, a potential candidate for renewable energy generation, since the feedstock is widely available and very cheap. This study presents the production of bio-oil, char, and noncondensable biogas through flash pyrolysis by changing its process parameters. The biofuels were also examined to analyze their physical and chemical characteristics through various chromatographic techniques.

2. Materials and Methods

2.1. Materials. Wood and wood barks of *Albizia odoratissima* were collected in the city of west Coimbatore, India. The collected materials were initially tested to find their moisture level. The higher moisture content in the biomass was eliminated by pulverizing. The material was crushed to an average size of 0.5–0.75 mm. The sample was then dried under sunlight for three days. The dried samples were further kept in the furnace at 100°C. The samples were then tested for their moisture content and found to be less than 5%.

2.2. Experimental Set-Up. The experiments were conducted on a lab scale fluidized bed reactor that consists of a cylindrical tube of 50 mm ID and 100 cm long [31]. The reactor is heated externally with the help of an electrical heater. The heat input is controlled by the PID controller. The outlet pipe is connected to a cyclone separator where the escaped char content is collected. The evolved gas after char separation was allowed to condense into the condenser. The condenser is distributed with surplus water kept at 5°C. Five K type thermocouples have been attached at five different points. The feedstock to the reactor is permitted over the screw feeder. A sand particle of 0.5 mm in diameter is used for fluidization inside the reactor. The distributor plate, which is placed at the bottom, allows pressurized gas into the reactor bed. Initially, compressed air is admitted, and once the reactor has reached a desired temperature, the nitrogen gas is admitted more than minimum fluidization velocity.

2.3. Pyrolysis Experiment. Initially, the reactor was heated to reach the preferred temperature at the heating rate of 30°C/min. After that, the supply of air was cut and the N₂ was allowed to fluidize. The minimum fluidization velocity of the particle was estimated as 0.11 m/s. The experiments were conducted at different temperatures of 350–550°C at 50°C intervals. The experiments were further conducted by changing sweep gas flow rate and heating rate. For the analysis of sweep gas flow rate, the N₂ gas was supplied at 1.25, 1.75, 2.00, and 2.25 m³/hr by keeping the reactor at 450°C. The effect of heating rate was analyzed by changing heating rate as 20, 30, and 40°C/min. The biomass samples were supplied continuously at 25 g/min. In order to analyze the product distributions, the experiments were conducted at two different phases. The first phase deals with the effect of temperature, and the second phase deals with the effect of sweep gas flow rate. Each run was conducted till no vapour was physically identified from the reactor. The condensed bio-oil and char particles were collected and stored. For yield analysis, the obtained oil and char particles were weighed with the help of a digital weighing machine. The noncondensable gas fractions were found by remaining material balance [32].

2.4. Pyrolysis Reaction Mechanism. The following governing equations (1) to (3) represent the transformation of woody biomass into volatiles obtained from literature [33]

$$\frac{d\alpha}{dt} = f(T)f(\alpha), \quad (1)$$

$$f(T) = Ae^{-(Ea/RT)}, \quad (2)$$

where A is pre-exponential factor, R is universal gas constant (J/mol K), Ea is activation energy (kJ/mol), and T is absolute temperature

$$\alpha = \frac{m_i - m_a}{m_i - m_f}, \quad (3)$$

where α is mass fraction of conversion, m_i is initial mass, m_a is actual mass, and m_f is final mass. Under constant temperature the equations (1) and (2) can be rewritten as [34].

$$\frac{d\alpha}{dt} = \frac{A}{\beta} e^{-(Ea/RT)} f(\alpha), \quad (4)$$

where β is heating rate.

2.5. Characterization Study. The proximate analysis of the feedstock was done by the proximate analyzer. The amount of fixed carbon in the material was found by difference. The ultimate analysis of the biomass and char elements was found directly using a CHNS analyzer, while the amount of oxygen was found by difference. All the analyses were performed by following ASTM standards. The degradation study of the biomass was analyzed using the TGA701 analyzer (LECO Corporation, Michigan). The TGA was performed under an N₂ atmosphere by heating the samples to 700°C. FT-IR and GC-MS (THERMO GC, VER: 5.0, MS DSQ-II) spectroscopy were employed for chemical analysis of the bio-oil and gas. The physical analysis of oil was done by the standard Pensky Martein closed cup apparatus, Parr-6772 calorimetric thermometer, digital pH meter, and redwood viscometer.

3. Results and Discussion

3.1. Biomass Characterization. The results of the analysis of the biomass material are revealed in Table 1. It is understood that the material has higher volatile matter (61.3%). Bio-oil and biogas are produced in large amounts from biomass with increased volatile matter [35]. The presence of more fixed carbon (25.36%) yields more biochar and boosts the carbon conversion rate, resulting in the highest heating value. The elemental analysis of the biomass reveals that the material has higher carbon and hydrogen. The lower amount of nitrogen and sulphur is recommended to produce biofuel with low environmental effects.

3.2. Thermogravimetric Analysis. Differential thermogravimetric (DTG) and TGA were done to analyze the pyrolysis behaviour under the nitrogen environment. The analysis was carried out by heating 5 mg of powdered sample kept on an aluminium pan to 700°C at the rate of 20°C/min. According to the TG curve (Figure 1), the first thermal event occurred at 54°C resulting in the weight loss of 6.95%. This

TABLE 1: Characterization of biomass.

Parameter	Proximate analysis (wt%)			Ultimate analysis (wt%)								
	Volatile matter	Fixed carbon ^s	Moisture content	Ash	C	H	N	S	O ^s	H/C	O/C	Heating value
<i>Albizia odoratissima</i>	61.3	25.36	6.40	6.94	51.04	6.28	1.20	0.01	50.47	1.779	0.901	19.21

^sBy difference.

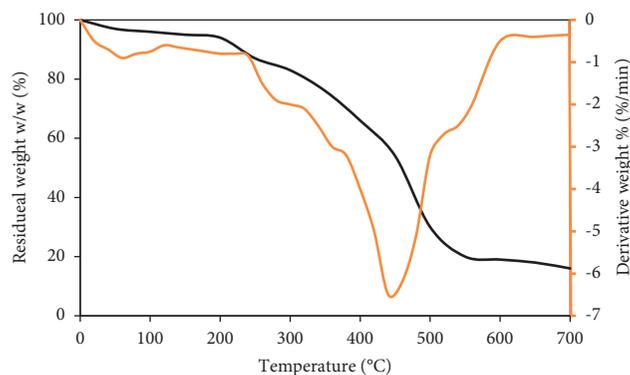


FIGURE 1: TG and DTG curve of the biomass material.

phenomenon is representing the evaporation of water molecules from the material [36]. The second peak appeared at 235°C attributed to the breakdown of lignocellulosic content. After 500°C, the samples were converted into carbon residues representing the breakdown of lignin [37]. After this stage, the passive pyrolysis occurs with leaving 18% of solid residues.

3.3. Pyrolysis Experiment

3.3.1. Impact of Temperature. Figure 2 represents the impact of process temperature on pyrolysis of biomass. Increasing temperature resulted in the maximization of bio-oil. It can be understood that the higher reactor temperature direct to maximum bio-oil production, while lower temperatures result in higher char production. Therefore, optimal bio-oil can be reached at 450°C (43.2 wt%). Increased temperature permits heavy hydrocarbon compounds to break, resulting in increased oil and gaseous content and lower char yield [38]. While increasing temperature from 450 to 550°C, the oil output was decreased by 9.72%. Horne and Williams [39] and Varma et al. [40] also reported a same trend related to pyrolysis temperature. The char produced during initial pyrolysis reactions underwent secondary reactions at elevated temperatures that resulted in increased gas products with reduced char. At elevated temperatures, more heat energy supplied to the material may surpass the bond cessation energy, releasing more volatiles. These volatile biomass elements are released as gases, leading to minimize the char and oil production. The gas yield at 350°C was 27.6 wt% and it reached to 40.2 wt% at 550°C. Due to a considerable loss of volatiles or secondary decomposition reaction at elevated temperatures, the production of char decreases. This may also result in the production of certain

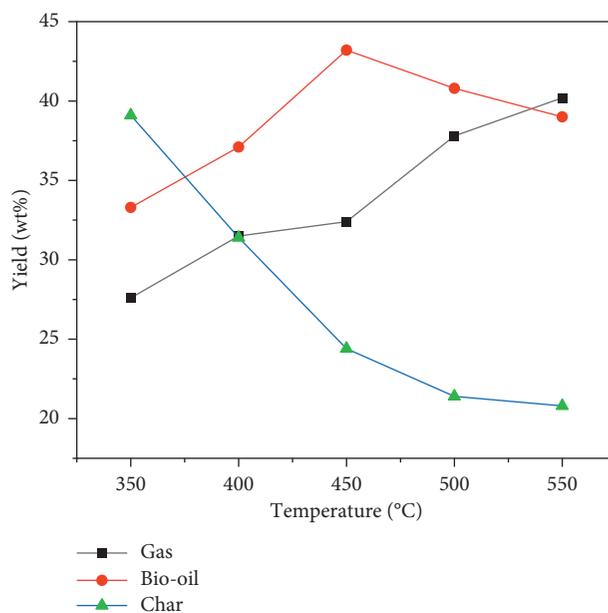


FIGURE 2: Effect of temperature on product distribution.

gases [41]. The yield of char in this study decreased from 39.1 to 20.8 wt% as the temperature was changed from 350°C to 550°C.

3.3.2. Effect of Nitrogen Gas Flow Rate. Nitrogen is generally used as a carrier gas for fluidized purpose and its flow rate is considerably affecting the yield of products [42–44]. The experiments for this phase were carried out at 450°C by varying the sweep gas flow rate. The vapours generated from the reactor at different N₂ flow rates were condensed into bio-oil, which is reported in Figure 3. From the figure, it is understood that the oil yield was increased up to 47.4 wt% when the flow rate is changed from 1.25 m³/hr to 1.75 m³/hr. At 1.75 m³/hr, the optimum oil output achieved a maximum of 47.4 wt%. The inadequate quenching of pyrolysis vapours in this phase increases the gas flow in this case. According to Uzun et al. [45], eliminating the developed pyrolysis vapours quickly with surplus N₂ and adequate quenching should result in higher bio-oil yields. With a continuous increase in sweep gas flow rate, the production of char is steadily increased from 24.4 wt% to 33.4 wt%. The higher flow rate of N₂ accelerated the movement of particles, resulting in good mixing of sand and biomass. The proper mixing of biomass and sand boosted heat transfer and resulted in increased condensable volatiles.

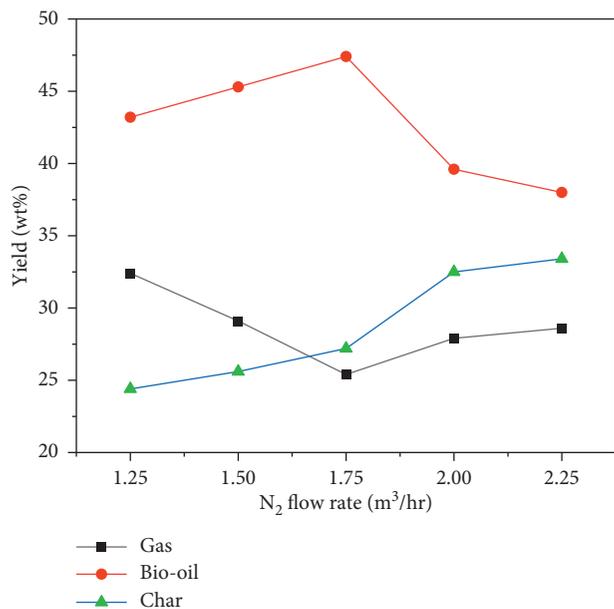


FIGURE 3: Effect of sweep gas flow rate on product distribution.

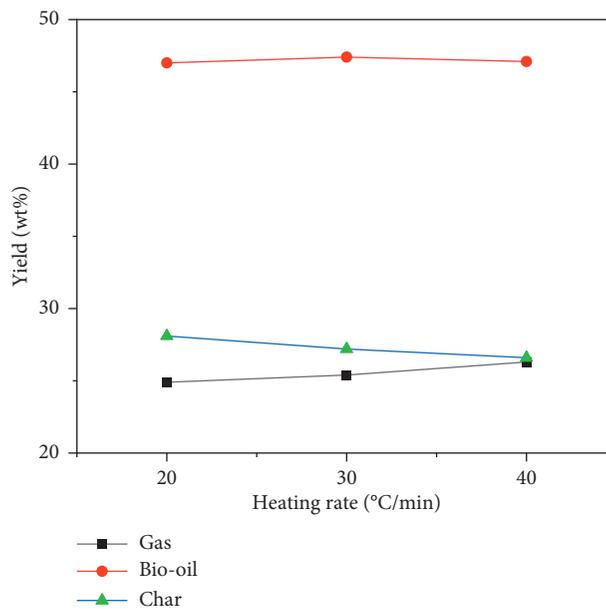


FIGURE 4: Effect of heating rate on product distribution.

3.3.3. Effect of Heating Rate. Heating rate is also an important factor that determines the product distribution. Many studies have conducted the effect of heating rate on biomass pyrolysis [46, 47]. Generally, the higher heating rate breakdowns the heat transfer restrictions and increases the release of volatiles. In this study, in comparison to the heating rate, temperature has a greater impact on pyrolysis yields [48]. There were no significant changes in pyrolysis product yields. With an increasing heating rate, the gas product is increased. At 20°C/min, the yield of gas was 24.9 wt% which is increased to 26.3 wt% at 40°C/min. The yield of char was reduced by 5.34% when the heating rate was increased. The yield of oil was increased at 30°C/min. At the point, the yield of oil was 47.4 wt% and the yield was decreased to 47.1 wt% at 40°C/min. The heat transfer limitations at higher heating rates increase the production of gas fractions [49]. Figure 4 represents the effect of heating rate on product distributions.

3.4. Product Characterization

3.4.1. Analysis of Bio-Oil. The basic characteristics of the bio-oil are displayed in Table 2. The physical analysis is a crucial one since the quality of the oil is determined by its physical characteristics. The application and suitability of the bio-oil are also identified based on its physical nature. The higher density and viscosity compared to petrodiesel shows that the oil affects the operation of the fuel atomizer during firing. The higher flash point represents safety during storage. The heating value is a vital one for any oil, which represents the burning efficiency which is found to be 18.15 MJ/kg. The bio-oil can be further processed using various chemical treatments to improve the heating value.

The functional group composition identified through FT-IR is shown in Figure 5. The large absorbance peak of

TABLE 2: Physical characteristics of the bio-oil.

Unit	Density kg/m ³	Viscosity cSt	pH —	Flash point °C	Heating value MJ/kg	Reference
Bio-oil	1020	7.2	3.6	145	18.15	This study
Diesel	850	3.9	—	57	43.6	[50]

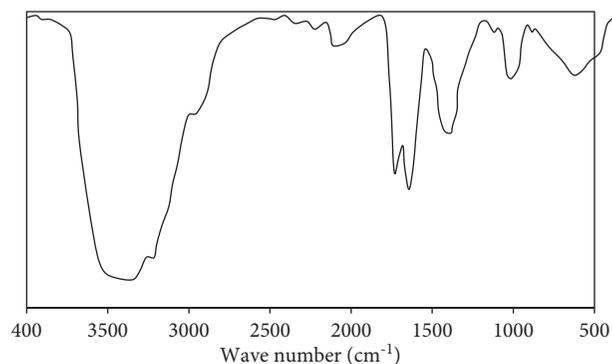


FIGURE 5: FT-IR analysis of the bio-oil.

O-H stretching vibration at 3450 cm⁻¹ suggested the presence of water, alcohols, phenols, and other hydroxyl compounds [51, 52]. Alkanes are identified by the absorbance peak of C-H vibration at 2950 cm⁻¹. The occurrence of ketones and aldehydes in the oil is pointed out by the C=O stretching vibrations at 1720 and 1680 cm⁻¹. The availability of alkane group components is identified by the CH bending at 1385 cm⁻¹ [53]. The presence of alcohols is further identified by the C-O stretching vibration at 1030 cm⁻¹ [54].

The chemical composition of the pyrolysis oil changes significantly depending on the nature of raw material and

TABLE 3: GC-MS analysis of the bio-oil.

Retention time (min)	Compound name	Formula	Area (%)
7.62	2-Isopropyl-2,5-dihydrofuran	C ₇ H ₁₂ O	3.72
7.56	2-Acetyl furan	C ₆ H ₆ O ₂	7.94
9.04	5-Hydroxymethylfurfural4	C ₆ H ₆ O ₃	1.78
12.36	Pyrogallol 1,3-dimethyl ether	C ₈ H ₁₀ O ₃	3.04
13.44	2,6-Dimethylphenol	C ₈ H ₁₀ O	4.07
17.99	2,3,5-Trimethoxytoluene	C ₁₀ H ₁₄ O ₃	3.14
18.42	2,5-Dimethoxytoluene	C ₉ H ₁₂ O ₂	2.11
18.56	3-Methylphenol	C ₇ H ₈ O	4.94
20.04	Tetradecane	C ₁₄ H ₃₀	2.65
20.55	Phenol	C ₆ H ₆ O	19.26
21.27	Di-(2-ethylhexyl) phthalate	C ₂₄ H ₃₈ O ₄	3.44
21.46	Phenol, 2,6-dimethoxy	C ₈ H ₁₀ O ₃	3.48
22.04	Eugenol	C ₁₀ H ₁₂ O ₂	1.50
22.38	Ethanol	C ₁₃ H ₂₂ O ₃	0.99
22.71	Phenol, 2-methoxy	C ₇ H ₈ O ₂	4.67
23.45	2-Isopropyl-2,5-dihydrofuran	C ₇ H ₁₂ O	6.41
24.11	2-Methoxy-3-methylhydroquinone	C ₈ H ₁₀ O ₃	2.97
24.87	Cyclopentanol	C ₅ H ₁₀ O	1.46
25.22	2,3,5-Trimethoxytoluene	C ₁₀ H ₁₄ O ₃	3.38
26.45	1,2-Benzendiol	C ₆ H ₆ O ₂	4.50
27.08	Nonane, 5-methyl-5-propyl-	C ₁₃ H ₂₈	3.28
28.50	1,2-Bis (2'-quinolylmethyl) ethylene	C ₂₀ H ₁₄ N ₂	1.86
29.41	Octacosane	C ₂₈ H ₅₈	2.93
29.49	Campesterol	C ₂₈ H ₄₈ O	3.83
29.78	Pregnenolone	C ₂₁ H ₃₂ O ₂	1.81

TABLE 4: Component analysis of char.

Proximate analysis (wt%)			Ultimate analysis (wt%)						H/C	O/C	Heating value (MJ/kg)
Volatile matter	Fixed carbon ^s	Moisture content	Ash	C	H	N	S	O ^s			
24.52	68.31	2.12	5.05	52.41	7.54	3.22	0.21	36.62	1.714	0.524	23.47

^sBy difference.

pyrolysis conditions [55]. During thermal breakdown, the primary components of feedstock, such as hemicelluloses, cellulose, and lignin are usually transformed into different hydrocarbons, phenols, and acids. Table 3 displays the outcome of the GC-MS analysis. The oil was verified to be a combination of various chemicals. The analysis showed a higher amount of phenols and their derivatives. The total area percentage of these compounds are 36.42. Phenol is an aromatic organic compound that is primarily used for various chemical processes. This is a very important chemical used for the production of nylon and other synthetic fibers. From this analysis, we can know that the chemical components identified in this study can be used for various chemical and pharmaceutical industries.

3.4.2. Analysis of Char. The characteristics of char components were determined and listed in Table 4. The heating value of the char is found as 23.47 MJ/kg. The percentage of carbon in the char is 52.41 wt%. On the other hand, the percentage of oxygen is identified as 36.62 wt%. The value of the carbon can be increased by increasing carbonization temperature. The higher carbon with lower oxygen can be recommended for use as a fuel. The lower amount of

nitrogen and sulphur in char is shown to be good for environmental sustainability during burning. For household cooking, the volatile matter should be between 20 and 40%. The volatile matter of the char is found to be within the range. The higher volatile content makes it easy to ignite although it can produce a smoky flame [56]. The fixed carbon is 68.31 wt%, which is in the range between 65 and 90% as recommended by Mythili and Venkatachalam [57]. The moisture level is identified as 2.12 wt%, which is much lower than the acceptable level of the charcoal (7%). The lower ash level of 5.05 wt% in the char implies that using it as a fuel may result in the production of lower clinkers.

3.4.3. Analysis of Noncondensable Gas. The noncondensable gases fractions are shown in Figure 6. Hydrogen is identified as a major one with 8.04%. Carbon dioxide and carbon monoxide are also detected at 6.47% and 2.14%, respectively, in the pyrolysis gas. The greater carbon monoxide than carbon dioxide is due to the reverse Boudouard reaction [58]. It is an important reaction inside the reactor, whereas the drop of iron oxides is not attained directly by carbon but by carbon monoxide. The produced carbon dioxide endures a Boudouard reaction with char. Generally, the variation in

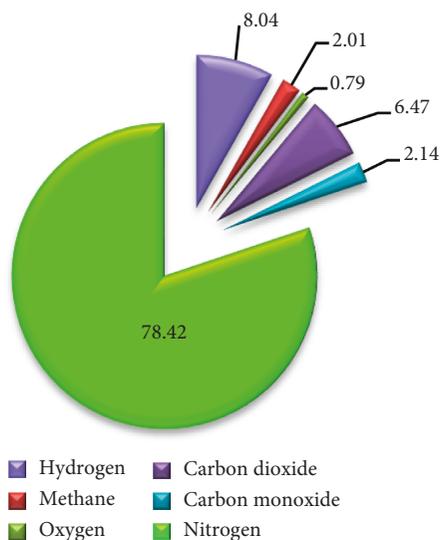


FIGURE 6: Noncondensable gas fractions.

hydrogen and carbon monoxide is a function of heat which is applied during pyrolysis [59]. The amount of methane in the gas is 2.01%.

4. Conclusion

Flash pyrolysis experiment on *Albizia odoratissima* has been performed at various pyrolysis temperatures and sweeping gas flow rates on a fluidized bed reactor. The selected wood sample yielded the maximum bio-oil of 47.4 wt% at optimized conditions of reactor temperature of 450°C sweeping gas flow rate of 1.75 m³/hr and heating rate of 30°C/min. The sample yielded maximum char and noncondensable gas of 39.1 and 40.2 wt%, respectively. The bio-oil produced in this study has a medium heating value, which can be further processed to be used as a fuel for industrial applications. The char has higher volatiles and fixed carbons with lesser ash and moisture, which makes it fit to utilize as a fuel for heating boilers and furnaces. The analyses of noncondensable gases showed that hydrogen and carbon dioxide were dominant, followed by carbon monoxide and methane.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this article.

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