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# Biofuel production of neem wood bark (*Azadirachta indica*) through flash pyrolysis in a fluidized bed reactor and its chromatographic characterization

C. Sowmya Dhanalakshmi  and P. Madhu 

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## ABSTRACT

In this study, wood bark of neem tree (*Azadirachta indica*) is transformed into liquid (bio-oil), solid (char) and gaseous products via pyrolysis method. The main aim of this study is to analyze the effect of process parameters on pyrolysis yield, physical and chemical characterization of bio-oil, char, and gaseous products to find its feasibility as a commercial fuel as well as chemical feedstocks. The flash pyrolysis experiments were carried out in a laboratory scale fluidized bed reactor at a temperature ranging from 350 to 550°C, different particle sizes from 0.71 to 1.25 mm with a sweep gas flow rate of 1.25 to 2.25 m<sup>3</sup>/hr. The highest liquid product yield was obtained as 49.5 wt% at the pyrolysis temperature of 450°C, 1.0 mm particle size and at the sweep gas flow rate 2.0 m<sup>3</sup>/hr. The obtained bio-oil, char, and gaseous products were examined with FT-IR, GC-MS and Elemental analysis methods. Based on the elemental analysis of the bio-oil, the calorific value was found as 22.7 MJ/kg. GC-MS analysis of the bio-oil indicates that it mostly consists of phenolic and oxygenated compounds with alkanes, alkenes, ketones, and carboxylic acids. The pyrolysis gas fraction contains hydrogen, methane, oxygen, carbon dioxide, and carbon monoxide. The obtained bio-oil and gas can be used as a bio-fuel and bio-oil can be used as a valuable chemical feedstock. The char obtained are carbon-rich and are nearly equal to the standard fuel properties.

## ARTICLE HISTORY

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## KEYWORDS

Wood bark; *Azadirachta indica*; pyrolysis; fluidized bed; biofuel; characterization

## Introduction

The increase in energy demands and the reduction of fossil fuel reserves along with environmental concerns have made it urgent to look for alternative and renewable energy sources (Durak and Genel 2018). Biomass has drawn great attention as a clean and alternative energy source emitting relatively low CO<sub>2</sub> levels, and a negligible amount of sulfur (Durak 2018). Biomass is a complex material, mainly consists of cellulose, hemicellulose, lignin, and inorganic salts. It can be transformed into bio-oils or high calorific value products via thermochemical processes such as liquefaction, pyrolysis, and gasification. The bio-oil is a promising contestant to replace fossil fuels (Madhu, Manickam, and Kanagasabapathy 2016). It can be used for the generation of heat, power and value-added chemicals. Pyrolytic bio-oil is known to be acidic, viscous, thermally unstable and contains high amounts of oxygenated compounds (Madhu, Livingston, and Kanagasabapathy 2018). Char or pyrolyzed charcoal is the secondary product obtained from the pyrolysis. It is a fine-grained porous product. Wood, agricultural crops and their residues are the common sources of biomass. Char or pyrolyzed charcoal is the secondary product obtaining from the pyrolysis. It is a fine-grained porous

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product. It is a main industrial fuel, which can be used for the production of activated carbon and other value-added chemicals. When it is applied as a soil amendment, it removes net carbon dioxide from the atmosphere. When added with fertilizer, it provides an improved habitat for soil organisms by improving nutrient retention (Yu, Steele, and Ruan 2010). The properties of the char are highly variable and depend on the type of raw biomass material and pyrolysis conditions. Different researchers have produced bio-oil products from wood, seeds, stems and de-oiled cakes by pyrolysis. Fast pyrolysis of pine bark and oak bark has been conducted by Ingram et al. (2008). The bio-oil produced from pine bark is having the heating value of 18.7 MJ/kg which is equal to that of bio-oil produced from pine wood. Whereas, the heating value of the oak bark pyrolysis oil is 19.0 MJ/kg which is more than that of bio-oil derived from oak wood. The literature suggested that both the bio-oils can be used as a low-grade bio-fuel. *Glycyrrhiza glabra* L. was transformed into liquid and solid products with catalysts ( $H_3BO_3$ , ZnO) and without catalyst at 250°C, 300°C, and 350°C by using the hydrothermal liquefaction method (Durak 2019). During this research, the highest conversion rate was obtained as 35.73 wt% in catalytic pyrolysis ( $H_3BO_3$  as a catalyst) at 350°C. According to the elemental analysis, the energy values of bio-oil products ranged between 21.87 and 31.78 MJ/kg and the energy values of solid products ranged between 16.22 and 18.28 MJ/kg. Şensöz (2003) conducted the slow pyrolysis experiment on pine bark (*Pinus brutia* Ten.). The effects of temperature, particle size, feed rate, sweep gas flow rate and heating rate on the yields and compositions of the bio-oil were investigated by various researchers. Durak, Genel, and Tunç (2019) conducted the pyrolysis experiments on black cumin seed cake and transformed into liquid and solid products. According to their results, the liquid products obtained at the end of the experiments are having higher heating values between 32.44 and 36.19 MJ/kg. The GC-MS analysis of the seed cake oil contains almost 70 pieces of compounds consisting of monoaromatics, aliphatics, oxygenated compounds with different properties. Nayan, Kumar, and Singh (2013) studied the pyrolysis behavior of neem seed in semi-batch reactor to determine particularly the effects of pyrolysis temperature and heating rate on the yield and their chemical compositions. The maximum bio-oil yield of 38 wt% was obtained at the temperature of 475°C at the heating rate of 20°C/min. Depending upon the operating conditions, the pyrolysis can be classified into conventional slow pyrolysis, fast and flash pyrolysis. Conventional slow pyrolysis can be suitable for the production of char which is performed at a lower heating rate with longer residence time. Fast and flash pyrolysis systems are well suitable for the production of liquid fuels. The quantity of liquid, char and gas products depends very much based on the pyrolysis techniques used on the reaction parameters. Due to the longer residence time, slow pyrolysis is not attractive for commercial applications of liquid fuel production. Nowadays the preferred technology for the liquid fuel production is fast or flash pyrolysis at a higher temperature around 400–800°C and at shorter residence time (Demirbas 2004). Pyrolysis of *Xanthium strumarium* has been performed by Durak (2016) in a fixed-bed tubular reactor with boron minerals at three different temperatures ranging from 350°C to 550°C with a heating rate of 50°C/min. In this research, pyrolysis temperature is found to be the main factor affecting the conversion into solid, liquid, and gaseous products.

*Azadirachta indica* is commonly known as neem tree. It is a tree in the mahogany family Meliaceae. It is a species of genus *Azadirachta* native to Indian subcontinents. It is naturally grown in tropical and semi-tropical regions. It is the most valuable tree that has increased worldwide significance due to its multiple uses. Neem trees are considered to be a marvelous tree in India because of their abundant valuable uses. The tree is used in multiple ways to the human. The leaves are dried and placed in cupboards to prevent insects. Neem products are believed by Siddha and Ayurvedic medical practitioners to be antifungal, antibiotic, antibacterial, antiviral, anthelmintic, contraceptive, and sedative. The neem wood has been separated from its bark and is mainly used for carpentry works. The wood bark is bulky to carry, it has a poor shelf life. Since there is no market value, these barks are used for cooking purposes. The open burning of these barks has a severe effect on the environment. So proper recycling is essential and it should be identified. The literature

available for wood bark energy conversion technique is minimum and no work has been done on neem bark pyrolysis system.

This work focuses on pyrolysis characteristics of wood barks of *Azadirachta indica* for the production of biofuels. Fluidized bed flash pyrolysis of the samples were carried out with different temperature ranges from 350 to 550°C, different particle sizes from 0.71 to 1.25 mm with a sweep gas flow rate of 1.25 to 2.25 m<sup>3</sup>/hr in order to examine their effect on pyrolysis yield. The effect of various parameters on pyrolysis yield and volatiles were also studied. The obtained biofuel products were characterized for different physical and chemical properties using FT-IR and GC-MS to find out the suitable applications.

## Method

### Materials

The wood barks were collected from Tirunelveli, India. The barks were crushed, dried in open atmosphere as well as in a vacuum oven at 40°C. The samples were ball-milled and separated by sieve shaker to obtain four different sizes- 0.6, 0.71, 1.0 and 1.25 mm. Table 1 shows the proximate, ultimate and lignocellulosic content of the wood bark sample and obtained char. The ultimate analysis of the biomass sample was performed by using Vario EL-III, Germany Elementar analyzer. Traditional wet chemistry method is adopted to find out the lignocellulosic content of the sample and it measures the weight difference after treatments using KOH and H<sub>2</sub>SO<sub>4</sub>. The sample contains more fractions of fixed content and volatile matters which gives a hope for more biomass conversion rate. The biomass samples may be environmental friendly due to its low fractions of nitrogen and sulfur.

### Reactor setup

The fluidized bed reactor used for the present study is the same as that used in a previous paper by the literature (Madhu, Manickam, and Kanagasabapathy 2015) and further is explained in this section. The reactor consists of a stainless steel tube of 1.0 m height with 50 mm inner diameter. It was heated by using 2 kW electrical resistance heater with ammeter and voltmeter setup. K type thermocouples located at five different points are used to measure the temperature of the reactor. The temperature inside the reactor was controlled by a PID controller. The reactor is insulated with mineral wool and Chromel–Alumel. Nitrogen gas is used as an inert gas for fluidization. Initially,

**Table 1.** Proximate and ultimate analysis of the biomass and char.

Parameters	Biomass	Char	Standard
Proximate analysis in wt%			
Volatile matter	69.8	23.13	ASTM D3175
Fixed Carbon	15.7	66.39	By difference
Moisture Content	10.3	4.36	ASTM D3173
Ash	4.2	6.12	ASTM D3174
Ultimate analysis <sup>a</sup> in wt %			
Carbon	41.51	41.51	ASTM D5373
Hydrogen	7.30	7.30	ASTM D5373
Nitrogen	5.41	5.41	ASTM D5373
Oxygen	45.36	45.36	By difference
Sulfur	0.42	0.42	ASTM D5373
H/C molar ratio	2.096	2.096	-
O/C molar ratio	0.821	0.821	-
Empirical formula	CH <sub>2.9</sub> N <sub>0.11</sub> O <sub>0.82</sub>	CH <sub>2.9</sub> N <sub>0.11</sub> O <sub>0.82</sub>	-
Lignocellulosic content in wt%			
Cellulose (± 0.05%)	17.58	-	-
Hemi-cellulose (± 0.05%)	42.56	-	-
Lignin (± 0.05%)	39.86	-	-

<sup>a</sup>Dry ash basis

compressed air was admitted till the reactor bed reaches the desired temperature and then the nitrogen gas replaces the air. The nitrogen flow rate was measured with the help of rotameter attached outside the reactor. The fluidizing gas velocity was maintained two times greater than the minimum fluidization velocity of 0.11 m/s. The nitrogen flow rates used for this study are 1.25, 1.75, 2.0 and 2.25 m<sup>3</sup>/hr. The sand particle used for all the experiments was 0.75 mm. The biomass samples were fed into the reactor through screw feeder which is attached at the height of 600 mm with the reactor through feeding port. When the reactor reaches the desired pyrolysis temperature, the samples were into the reactor at the rate of 30 g/min. The released volatile compounds from the reactor were quenched by the condenser by the flow of cooled water maintained at 10°C. The condensed bio-oil was collected in a flask and weighed. The char was collected from the bottom of the reactor and weighed. The conversion of biomass sample to bio-fuels on weight ratio was calculated by following the total amount of bio-oil collected per batch/total amount of biomass sample fed) × 100. The yield of char was calculated by (the total amount of char collected/total amount of biomass sample fed) × 100. The uncondensable gas released during the reaction was calculated by the overall material balance.

### Experimental procedure

The aim of the first series of the experimental work is to find out the effect of process temperature on pyrolysis yield. For this, the experiments were conducted at different temperatures of 350, 400, 450, 500, 550°C at the constant particle size of 0.71 mm and at the sweep gas flow rate of 1.75 m<sup>3</sup>/hr. The second part of the experiment is conducted to determine the effect of particle size on pyrolysis yield. For this purpose the experiments are conducted with four different particle sizes of 0.6, 0.71, 1.0, 1.25 mm with the optimum pyrolysis temperate of 450°C and at the sweep gas flow rate of 1.75 m<sup>3</sup>/hr. In order to study the effect of sweep gas flow rate during pyrolysis reaction, the third series of experiments are conducted by varying the flow rate as 1.25, 1.75, 2.0, 2.25 m<sup>3</sup>/hr at the pyrolysis temperature of 450°C and at the particle size of 1.0 mm. All experiments were performed thrice and the average value was taken into consideration. The average yields are given within the error of less than ± 2 wt%. Table 2 shows the experimental conditions of the present study.

### Characterization methods

BROOKFIELD LV-DV-II Pro viscometer and Penskey Martein closed cup apparatus were used to measure the viscosity and flash point of the bio-oil sample. Parr-6772 calorimetric thermometer was used to measure the calorific value of the bio-oil. The density was measured by weighing the known volume of the sample. Digital pH meter was used to measure the pH value of the bio-oil. Elementar Vario EL-III instrumentations with WinWar software was used to measure the components of the bio-oil and char. Fourier transform infra-red spectroscopy (FT-IR) was used for the analysis of chemical investigations of

**Table 2.** Experimental conditions and pyrolysis yield.

Case	Temperature in °C	Particle size in mm	Sweep gas flow rate in m <sup>3</sup> /hr
Run 2	400	0.71	1.75
Run 3	450	0.71	1.75
Run 4	500	0.71	1.75
Run 5	550	0.71	1.75
Run 6	450	0.6	1.75
Run 7	450	0.71	1.75
Run 8	450	1.0	1.75
Run 9	450	1.25	1.75
Run 10	450	1.0	1.25
Run 11	450	1.0	1.75
Run 12	450	1.0	2.0
Run 13	450	1.0	2.25

**Table 3.** GC-MS conditions.

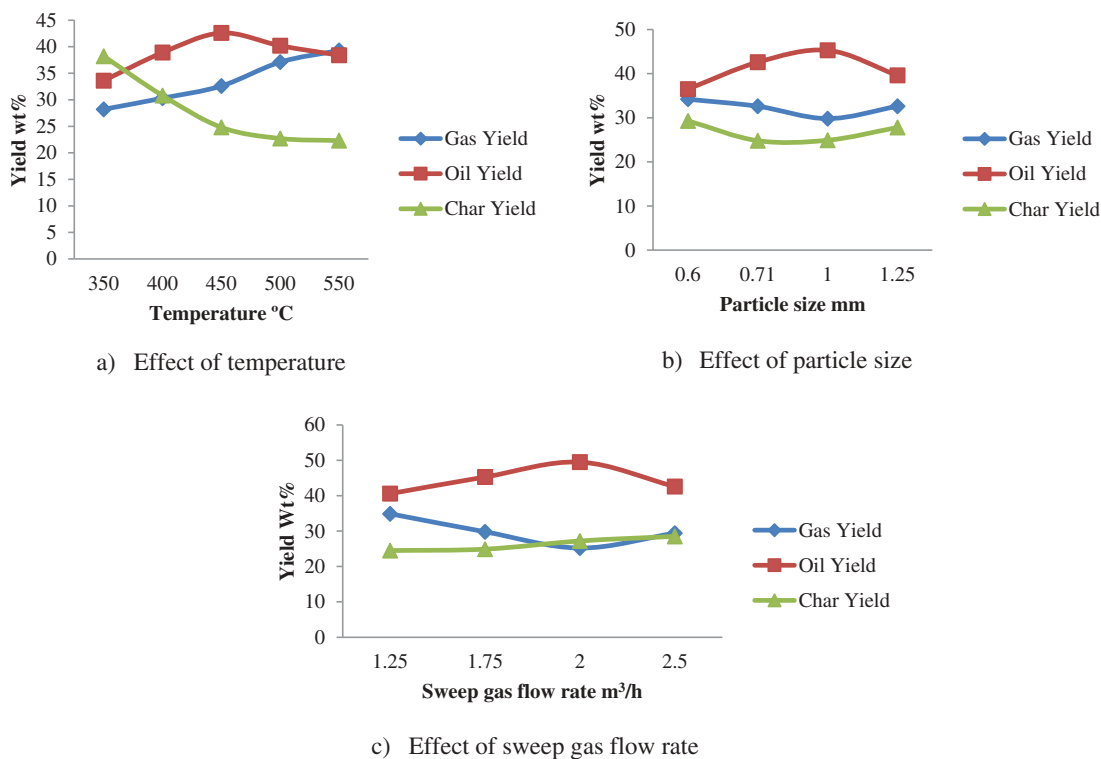
Instrument	Thermo MS DSQ II	
GC conditions		
Column oven temperature	70°C	
Injection mode	Split	
Injection temperature	200°C	
Spit ratio	10	
Flow control mode	Linear velocity	
Column flow	10 ml/min	
Carrier gas	Helium	
Column oven temperature progress		
Rate	Temperature in °C	Hold time in m
-	70	5
10	250	7
Column DB-35		
Length	30 m	
Diameter	0.25 mm	
Film temperature	0.25 µm	
MS Conditions		
Ion source temperature	200°C	
Interface temperature	250°C	
Start <i>m/z</i>	50	
End <i>m/z</i>	650	

the bio-oil. For this Bruker Optik GmbH Tensor27 by Opus version 6.5 was used at resolution  $4\text{ cm}^{-1}$  among  $4000\text{ to }400\text{ cm}^{-1}$ . Thermo MS DSQ II Gas chromatography–mass spectrometry was used for the analysis of volatile and semi-volatile of the bio-oil and gas. The GC conditions, column oven temperature, the column used, and MS conditions are given in Table 3.

## Results and discussion

### *Effect of temperature on product yield*

During pyrolysis, biomass undergoes end to end primary and secondary reactions relating heat and mass transfer mechanism. Decomposition of cellulose, hemicelluloses, and lignin present in the biomass occurs primarily which leads to the formation of primary products and intermediates. These intermediate species further undergoes secondary cracking. The basic function of temperature is to supply necessary heat for the decomposition of biomass linkages. The pyrolysis experiments were conducted by varying the temperature values (350, 400, 450 500 550°C) at a constant particle size of 0.71 mm and at constant sweep gas ( $\text{N}_2$ ) flow rate of  $1.75\text{ m}^3/\text{hr}$ . Obviously, the optimum temperature in this condition for maximum bio-oil is in the range of 450°C. Figure 1 shows the variation of percentage mass of bio-oil, char and gas products in relation to the mass of biomass feed at different bed temperatures, different particle sizes, and different sweep gas flow rates. When the temperature increases from 350 to 550°C, the yield of bio-oil increases from 33.6 wt% to 36.4 wt%. The result shows that as the reactor temperature was increased the bio-oil yield also increases up to 450°C. After this temperature, the bio-oil yield was decreasing. On the other hand, the yield of char is reduced from 38.2 wt% to 24.3 wt% and the gas yield is increased from 28.2 wt% to 39.3 wt%. At a lower temperature of ( $<350^\circ\text{C}$ ), the bio-oil yield was only 33.6 wt% with a higher char yield of 38.2 wt%. The decomposition of the feed particle at a lower temperature which occurs at heteroatom within the structure gives more char yield (Tsai, Lee, and Chang 2007). At an elevated temperature, the massive breakup of biomass material causes high molecular dislocation and produces various types of chemical components. When the temperature is maintained less than 350°C, the conversion efficiency of biomass into bio-oil is very low. Huge conversion of biomass to bio-oil and its fragments occur within the temperature range of 400–500°C. Increase of temperature gives additional energy to break the biomass structure as the conversion efficiency is increased. At a pyrolysis temperature of 450°C, because of strong cracking the yield of bio-oil will be a maximum of 42.6 wt% and the yield is



**Figure 1.** Effect of process parameters on pyrolysis yield: (a) effect of temperature, (b) effect of particle size, and (c) effect of sweep gas flow rate.

decreased to 36.4 wt% when the temperature is beyond 450°C due to the secondary cracking reactions of the pyrolysis vapor. According to Sohaib, Muhammad, and Younas (2017) with the increase in pyrolysis temperature, both bio-oil and gaseous yields are increased due to the secondary decomposition of char residues. Many researchers also studied the effect of temperature on product yields to establish the optimum temperature for maximum bio-oil yield. The yield of palm shell was reported by Abnisa et al. (2011) who confirmed the yield of 47.3 wt % at 500°C. Pütün, Özcan, and Pütün (1999) reported that 500°C as the maximum yield temperature for hazelnut shells with a yield of 23.1 wt%. Similarly, Madhu, Livingston, and Kanagasabapathy (2018) reported that at a temperature of 450°C, the maximum bio-oil yield of 50.6 wt% was obtained from the flash pyrolysis of lemongrass.

### **Effect of particle size on product yield**

Generally, the useful size of the feed particles may vary depending upon the type of biomass and type of pyrolyzer. Understanding the effect of particle size during pyrolysis product distributions can be explained by the conductivity of the samples. Biomass being poor thermal conductor poses heat transfer difficulties during pyrolysis. The yield of pyrolysis products depends on the size of the feed particles. The bio-oil yield is significantly increased and the gas yield is decreased when the size of the particle is increased from 0.6 mm to 1.0 mm. The result shows that the bio-oil yield is increased from 36.5 wt% to 45.3 wt%. On the other hand char and gas yield is decreased from 29.3 wt% to 24.9 wt% and 34.2 wt% to 29.8 wt%, respectively. The yield of bio-oil yield is decreased to 39.6 wt%, when the particle size is increased from 1.0 mm to 1.25 mm. Generally, smaller particle sizes are preferred in pyrolysis systems as they can heat up uniformly than larger particles. The poor heat and mass

transfer restrictions to the inner surfaces of the larger particles causes low average particle temperatures which yields lower bio-oil and the particle size of 1.0 mm is suitable for the production of higher bio-oil yield. Based on these results, particle size has an important parameter during pyrolysis product distributions. Biomass structure is another important parameter to explain the product distributions. Cell structure may affect the pyrolysis behavior, such as the release of alkaline earth metallic species.

### **Effect of sweep gas flow rate on product yield**

To find out the effect of sweep gas flow rate on pyrolysis product yields, the experiments were conducted at four different sweep gas flow rates of 1.25, 1.75, 2.0, 2.25 m<sup>3</sup>/hr. The pyrolysis temperature and particle size for this set of experiments were kept constant as 450°C and 1.0 mm, respectively, which is obtained from the optimization of the first and second group of experiments. The yield of bio-oil is increased from 40.6 wt% to 49.5 wt% when the flow rate of nitrogen is increased from 1.25 to 2.0 m<sup>3</sup>/hr and further the yield is decreased to 42.6 wt% when the flow rate of nitrogen is increased to 2.25 m<sup>3</sup>/hr. At the same time, the yield of gas is decreased from 34.9 wt% to 25.2 wt%. With an increase in the gas flow rate from 1.25 m<sup>3</sup>/hr to 2.25 m<sup>3</sup>/hr, a continuous increase in the char yield has been observed from 24.5 wt % to 28.5 wt%. From this study, a maximum bio-oil yield of 49.5 wt % was obtained at the sweep gas flow rate of 2.0 m<sup>3</sup>/hr. According to this study, the fluidization condition and vapor residence time directly affect the yield of bio-oil, char, and gases (Dhyani and Bhaskar 2018). The sweep gas flow rate amplified the movement of the biomass particles inside the reactor and the mixing between the sand material and biomass becomes vigorous. The enhanced mixing of biomass with sand particles improves the heat transfer rate which enhances the volatile release. But the shorter residence time leads to the lower prospects of the secondary tar cracking. In this work, a maximum yield of bio-oil, char, and gas of 49.5, 28.5 and 34.9 wt% were attained at the sweep gas flow rate of 2.0, 2.25 and 1.25 m<sup>3</sup>/hr respectively.

### **Product characterization**

#### **Characterization of the bio-oil**

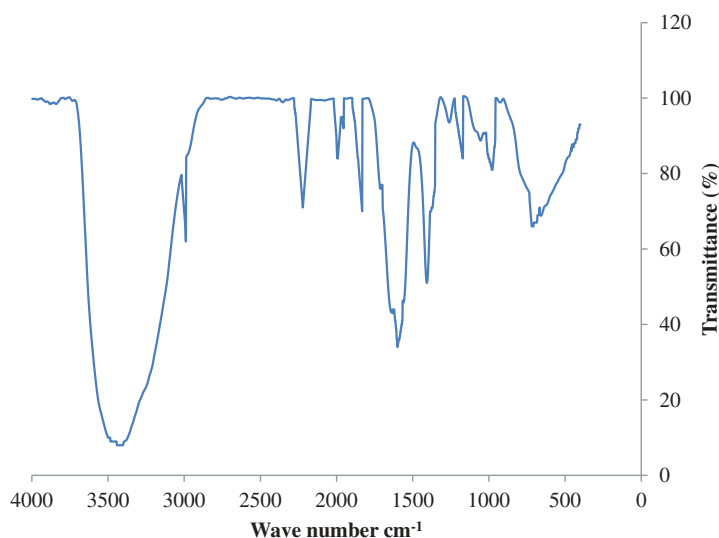
Table 4 shows the comparison of the physical properties of neem bark and other pyrolysis bio-oils obtained from different biomass materials and diesel. The pyrolysis bio-oil obtained from neem bark is highly oxygenated with a maximum oxygen content of 41.01 wt%. The obtained bio-oil has two layers, the upper layer is known as the fuel layer, and the bottom layer is known as the oily water layer. The bottom layer is a mixture of water and carbohydrate derived organic liquid which is the source of oxygenated compounds present in the biomass sample. The higher flash point of the bio-oil indicates that it can be stored safely at room temperature. The presence of acidic acid in the bio-oil was confirmed by the lower pH value. Since it can be corroded with steel, aluminum and nickel-based materials, it should be treated before any specific applications. The calorific value of the bio-oil is 22.7 MJ/kg which is good enough for the bio-oil produced from an agricultural waste. The higher density of the pyrolytic bio-oil can affect the fluid atomizer and it can be reduced by blending with some other transportation fuels.

Figure 2 shows FT-IR spectrum of the bio-oil. The bio-oil contains different organic compositions including aromatic, aliphatic and oxygenated functions. In the FT-IR spectrum, OH stretching vibration band was monitored between 3400–3200 cm<sup>-1</sup> indicate the polymeric hydroxyl compounds and alcohols in the bio-oil. The C-H bonds between 3000–2850 cm<sup>-1</sup> indicate the presence of alkyls. The absorbance peak between 2260–2240 represents C ≡ N stretch indicates nitriles in the bio-oil. The C-C stretching between 1600–1585 cm<sup>-1</sup> indicates the aromatics. The –CH<sub>2</sub> bending between 1445 and 1400 cm<sup>-1</sup> indicates the alkanes group in the bio-oil. The C-O absorbance peak between 1375–1000 cm<sup>-1</sup> indicates the presence of alcohols, carboxylic acids, esters, ethers, and the absorbance peak between 1000 and 650 cm<sup>-1</sup> represents = C-H bending vibrations indicates the alkanes.



**Table 4.** Comparison of properties of bio-oil.

Properties	Neem bark [This study]	Cotton shell (Madhu, Kanagasabapathy, and Manickam 2016)	Palm shell (Abrisa et al. 2011)	Albizia amara wood (Sowmya Dhanalakshmi and Madhu 2018)	Hard wood (Solantausta et al. 1993)	Diesel (Islam, Zailani, and Ani 1999)
Density (kg/m <sup>3</sup> )	1015	1005	1051	1050	1220	780
Viscosity (cSt)	8.1	7.87	3.2	4.2	13	1.3–3.3
Flash point (°C)	155	160	a	160	66	75
pH	3.7	3.3	2.5	3.6	a	a
Carbon (wt%)	48.82	38.14	19.48	47.54	55.5	86.58
Hydrogen (wt%)	9.1	11.24	8.92	7.8	6.7	13.29
Nitrogen (wt%)	0.84	0.94	0.2	0.5	0.1	65 ppm
Sulfur (wt%)	0.23	0.11	0.04	0.1	0.0	0.11
Oxygen (wt%)	41.01	49.57	71.40	44.06	37.7	0.01
H/C molar ratio	2.223	3.511	a	a	a	a
O/C molar ratio	0.630	0.975	a	a	a	a
Empirical formula	CH <sub>2.22</sub> N <sub>0.014</sub> O <sub>0.63</sub>	CH <sub>3.511</sub> N <sub>0.021</sub> O <sub>0.975</sub>	a	a	a	a
Heating value (MJ/kg)	22.7	19.32	6.58	18.63	17.5	45–46



**Figure 2.** FT-IR spectrum of the bio-oil.

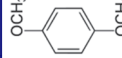
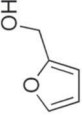

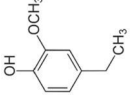
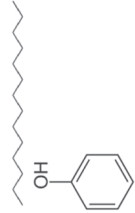


The compounds present in the bio-oil were known by comparing the chromatogram obtained with standard chromatogram data available in the library. From GC-MS analysis it has been observed that the bio-oil product contains the mixture of various compounds such as phenolics and other aromatic compounds. Cellulose and hemicelluloses of the biomass material are the sources of these aromatic and oxygenated compounds. Different types of compounds such as phenols, alkanes, alkenes, fatty acids, and esters were identified. Phenol, trimethylamine, 2-methylphenol, octadecanenitrile and stearic acid, methyl ester are identified with higher peak area the total area percentage of the phenol and its derivatives are 30. Phenols are used in the synthesis of antioxidant compounds. The main uses of phenol, consuming two-thirds of its production, involve its conversion to precursors for plastics. Octadecanenitrile is used as a chemical intermediate for fatty amines and derivatives. Trimethylamine is a colorless, flammable tertiary amine. At low concentrations, it has a strong fishy odor and has an ammonia-like odor at higher concentrations. It is used in the synthesis of choline, herbicides, tetramethylammonium hydroxide, resins, dye leveling agents and a number of basic dyes. Table 5 shows the various chemical compounds present in the bio-oil with its properties and uses.

### **Characterization of char**

The volatile matter present in the char is 23.13 wt%. According to Antal and Grønli (2003) it is a measure of carbonization that is directly proportional to the production temperature. Normally the volatile matter present in the char can vary from 40 to 5 % or less. The volatile matter content of the char for the domestic cooking is recommended as 20–40%. The char produced in this study has reasonable volatile matter content. The higher volatile matter present in the char leads easy ignition but may burn with a smoky flame (Misginna and Rajabu 2014). According to Mythili and Venkatachalam (2015) the fixed carbon content in the char may vary from 50 to 95%. As reported by Antal and Grønli (2003) the fixed carbon content in the charcoal may in the range of 65–90%. The fixed carbon present in the char is 66.39 wt%. By increasing the carbonization temperature, the fixed carbon content in the char can be increased. The moisture content present in the char is 4.36 wt% which is lesser than the recommended (<7%) moisture content of the charcoal. According to McLaughlin et al. (2009) the particular biomass used can affect the characterization and usability of the char. The char has the ash content of 6.12 wt% which is slightly higher than the prescribed limit which means that the usage of these char as a fuel, generates



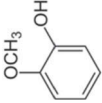
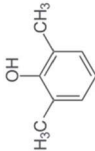
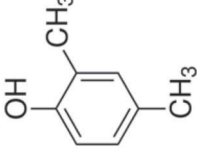
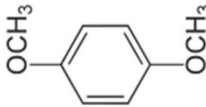


Table 5. GC-MS analysis of the bio-oil.

Compound Name	Molecular Formula	Molecular structure	% Area	Properties and uses
1,4-Dimethoxybenzene	$C_8H_{10}O_2$		1.26	1,4-Dimethoxybenzene naturally occurs in willow, tea and hyacinth. It is mainly used in perfumes and soaps. 1,4-Dimethoxybenzene is also used as an intermediate in synthesis of organic compounds in pharmaceutical industries. It can be used as a developer agent in black and white film.
2-furanmethanol	$C_5H_6O_2$		0.20	2-furanmethanol containing a furan with hydroxymethyl group. It is a colourless liquid, odor and bitter taste. It is miscible and unstable in water. It can be used in all organic solvents. Furanmethanol can be used in rocketry fuel that can ignite immediately. It is also used as a resin for thermoset polymer matrix composites, cements, adhesives. Due to its lower molecular weight, it can saturate the wood cells, where it can be bonded with wood by heat and catalytic reactions. Tridecane is a colourless combustible liquid. In industries it can be used as a fuels and solvent. This is an alkane hydrocarbon used in the manufacture of paraffin products, jet fuel research, and paper processing and rubber industries.
Tridecane	$C_{13}H_{28}$		0.81	
2-Isopropyl-2,5-dihydrofuran 4-ethyl-2-methoxy-phenol	$C_7H_{12}O$ $C_9H_{12}O_2$		3.75 1.55	
Pyrogallol 1,3-dimethyl ether n-Tetradecane Phenol	$C_6H_{10}O_3$ $C_{14}H_{30}$ $C_6H_6O$		0.99 1.52 12.62	Phenol is a volatile, crystalline aromatic organic compound. It was first extracted from coal. It is a very important precursor for many industrial products (Weber, Weber, and Kleine-Boymann 2004). Primarily it is used to synthesize plastic products. Its chemical derivatives are very important for the production of detergents, nylon, polycarbonates and various pharmaceutical drugs. Phenols are soluble significantly in water. It is well reactive to aromatic substitutions. The main uses of phenol consuming two third of its production entails its conversion to precursors for plastics. It also a precursor to a collection of drugs such as aspirin and herbicides. Medicinally phenol was used as an antiseptic and its spray is used to reduce throat pain. 2-methylphenol also called as o-Cresol. It is a colourless solid that is used as an intermediate in the production of some chemicals. It is a derivative product of phenol. The main uses of 2-methylphenol is a precursor to other compounds.
2-methylphenol	$C_7H_8O_2$		7.80	
3-methylphenol	$C_7H_8O$		2.66	3-methylphenol is commonly known as m-cresol extracted from coal. It is a volatile material acquired from the production of coal. The residue of m-cresol contains little weight percentage of isomeric cresols and phenol.

(Continued)



Table 5. (Continued).

Compound Name	Molecular Formula	Molecular structure	% Area	Properties and uses
2-methoxyphenol	$C_8H_8O_2$		1.22	2-methoxyphenol is also known as Guaiacol which is occurring naturally. It is yellowish aromatic liquid is typically derived from guaiacum or wood creosote. Guaiacol will be darken when it expose to air and light. Normally it is present in wood smoke obtained from the pyrolysis of lignin. It is used as aprecursor to various flavorants such as vanillin and eugenol (Esposito et al. 1997). Almost 85% of the vanillin derived from guaiacol. Whisky and roasted coffee has been flavored by these compounds. Generally, methoxyphenols are possible biomarkers of biomass smoke revelation, such as from inhalation of wood smoke. Glyoxylic acid will be condensed with guaiacol gives mandelic acid, which is oxidized to produce phenylglyoxylic acid. This will undergo a decarboxylation to give vanillin.
2,6-dimethylphenol	$C_8H_{10}O$		1.96	2,6-dimethylphenol is commonly known as 2,6-Xylenol. It is one of the six isomers of xyleneol. It has flash point of 165°C with boiling point of 203°C. It is used in the synthesis of Mexiletine. Its derivatives are used as food additives, flavoring agents, automotive care products, paints and coatings.
2,4-dimethylphenol	$C_8H_{10}O$		3.54	2,4-dimethylphenol is oily liquid or volatile colourless solid. It is a derivative of phenol with two methyl and hydroxyl group. Jointly with cresylic acid and cresols it is an very important group of phenolics with enormous applications. 2,4-dimethylphenol is used in the manufacture of antioxidants. It is a monomer for poly engineering resins.
1,4-dimethoxy benzene	$C_8H_{10}O_2$		0.81	1,4-Dimethoxybenzene produced by several plant species. It is white solid with sweet floral odor. It is one of three isomers of dimethoxybenzene. Naturally it is available in tea, willow, hyacinth and zucchini (Fahlbusch et al. 2003). It is main psychoactive chemical in musk willow. It can be used in perfumes, soaps and as an intermediate in synthesis of organic compounds. 1,4-dimethoxy benzene is widely used as a developer in black and white film.
Octadecanenitrile	$C_{18}H_{35}N$		7.68	-
Stearic acid, methyl ester	$C_{19}H_{38}O_2$		5.24	Stearic acid methyl ester is an esterified version of the free acid. It is less water soluble but agreeable for the formulation of dietary supplements. It is colourless clear liquid and that can be derived from vegetable oils and animal fats.

(Continued)



Table 5. (Continued).

Compound Name	Molecular Formula	Molecular structure	% Area	Properties and uses
Eugenol	$C_{10}H_{12}O_2$		0.63	Eugenol is a colourless clear aromatic oily liquid hauled out from essential oils such as clove oil, nutmeg, cinnamon, basil and bay leaf. It is a member of the phenylpropanoids class of chemical compounds. Eugenol naturally available in several plants that can be used as a local anaesthetic and antiseptic. It also used as a flavoring agent in perfumes and essential oils. Zinc oxide eugenol can be obtained by the combination of eugenol with zinc oxide which has wide dental applications such as surgical pastes, dental packing and dental cement.
5-hydroxymethylfurfural	$C_6H_6O_3$		0.10	5-hydroxymethylfurfural is a white low melting solid which is formed by the dehydration of some sugars (Van Putten et al. 2013). It is soluble more in water as well as organic solvents. The furan ring present in the molecule contains aldehyde and alcohol. 5-hydroxymethylfurfural can be converted to 2,5-dimethylfuran that is a prospective biofuel with a larger energy content than bioethanol.
Ethanol	$C_{13}H_{22}O_3$		2.12	Ethanol, also called ethyl alcohol, grain alcohol or simply alcohol. It is a volatile, flammable and colourless liquid with a slight odor. It is a psychoactive substance found in alcoholic drinks. Naturally it is produced by the fermentation of sugars by yeasts or by petrochemical processes. It is consumed as a common recreational drug. Medicinally it is used as an antiseptic - and disinfectant. Medical wipes, hand sanitizer gels have been produced by ethanol which gives anti-bacterial and anti-fungal effects (Pohorecky and Brick 1988). The largest single use of ethanol is as an engine fuel. It has been used as rocket fuel particularly for racing aircraft. Ethanol is a clean burning fuel source. During combustion in IC engines, the emission of CO, NOx, particulate matter and ozone-forming pollutants are very minimum. It is colourless liquid that has a slight odor. In open air, it burns with a smokeless blue flame that is not visible in normal sight. Ethanol is miscible with water and is a good solvent. It is found in tinctures, paints, markers, mouthwashes, perfumes and deodorants.
Kaempferol	$C_{16}H_{10}O_4$		3.27	Kaempferol is found in a variety of plants and pant derived foods. It is a natural flavonol, a type of flavonoid. It has melting point of 276–278°C, yellow crystalline solid which is soluble in water and highly can soluble in ethanol, esters. It is a derived metabolite originated in many plants (Calderon-Montano et al. 2011). Medicinally it acts as an antioxidant.
2,3,5-Trimethoxytoluene	$C_{10}H_{14}O_3$	-	2.25	1,2-benzendiol was first discovered by disparaging distillation of plant and wood extract catechin. It is a colourless compound occurs naturally. Mainly it is consumed in the production of perfumes, pesticides and is being used as a precursor to fine chemicals in pharmaceutical industries.
1,2-benzendiol	$C_6H_6O_2$		4.25	
Hexadecanenitrile	$C_{16}H_{31}N$		5.45	
Trimethylamine	$C_3H_9N$		8.22	Trimethylamine is a colourless, combustible and hygroscopic tertiary amine. It is product of decomposition of animals and plants. At lower concentration it has strong fishy odor and ammonia like odor at higher concentrations. It is used in the synthesis of herbicides, choline, plant growth regulators and dyes. It also used as a gas sensor due to its fishy odor.

(Continued)



Table 5. (Continued).

Compound Name	Molecular Formula	Molecular structure	% Area	Properties and uses
3-Acetoxy-24-methyl-5-cholest-5,22-dien-7-one	$C_{31}H_{48}O_3$	-	2.85	-
Stearic amide	$C_{18}H_{37}NO$	-	3.25	-
3,4-Alitrosan	$C_6H_{10}O_5$	-	3.10	-
9-Octadecenamide	$C_{18}H_{35}NO$	-	5.46	-
4-Fluorophenyl	$C_6H_5F$	-	2.26	-
Vanillin	$C_8H_8O_3$		0.79	Vanillin is a phenolic aldehyde. It is functional groups comprise aldehyde, hydroxyl and ether. It is the main component of the extract of the vanilla bean. 75% of the market for vanillin is used as a flavoring agent usually for sweet foods, ice cream and chocolate industries. It is also used in the fragrance industry, flavor industry. It is a very essential flavor for many food industries and for cream soda. It also can be used as a mask for unpleasant odor or tastes in medicines and cleaning products. Apart from these, it is also used as a stain for the development of thin layer chromatography plates. This stain gives a variety of different colours according to the components.
oleic acid	$C_{18}H_{34}O_2$		1.20	It is a fatty acid that naturally occurs in oils, animal and vegetable fats. Oleic acid is colourless and odorless. The main use of its derivatives is in the form of its triglycerides. It is consumed as a part of vegetable oil and animal fats in human diets. In pharmaceuticals, small amount of oleic acid are used as an excipient and in aerosol products it is used as a solubilizing agent.
Stigmast-5-en-3-ol, oleate	$C_{47}H_{82}O_2$	-	0.67	-

more clinkers that will negatively affect the system performance. During pyrolysis decrease in volatile matter content increase the percentage of fixed carbon. Table 1 shows the major variation in carbon and oxygen content and minor variation in hydrogen, nitrogen and sulfur content of the obtained char. The drastic decrease in volatile content indicates the higher conversion of biomass to bio-oil (Nayan, Kumar, and Singh 2013).

### Gas compositions

The major component of the pyrolysis gas comes out during pyrolysis were carbon monoxide and carbon dioxide. The percentage of CO<sub>2</sub> in the gas was in the range of 6.54 to 8.11% at the higher and lower temperature spectrum of 550°C and 350°C. At lower temperature, the percentage of the CO<sub>2</sub> is 7.17% and it is maximum at the temperature of 500°C. The percentage of CO<sub>2</sub> follows increase-decrease pater with increase the temperature. The concentration of CO is increased with increase in temperature upto 500°C and then it is decreased. Menendez et al. (2007) reported that the reverse boudouard reaction between the CO<sub>2</sub> and char may be the reason for increase in CO concentration at higher temperature. The concentration of CO was in the range of 2.07–3.45%. The concentration of Methane and hydrogen increased sharply with the higher temperature. The methane and hydrogen content was in the range of 0.12 to 2.26% and 1.62 to 7.92%, respectively. According to Kaupp (1984) the hydrogen and CO content in the gas are a strong function of temperature. Table 6 shows the various gas fractions at different operating conditions.

### Conclusion

In this study, flash pyrolysis experiments on Wood bark of neem tree (*Azadirachta indica*) were performed in a fluidized bed reactor under a nitrogen atmosphere which is a promising feedstock for the production of bio-fuel. During this pyrolysis, the highest bio-oil yield of 49.5 wt% was obtained at the temperature of 450°C with a particle size of 1.0 mm and at the sweep gas flow rate of 2.0 m<sup>3</sup>/hr. Reliable with the previous studies, the highest conversion is obtained at the temperature of 450°C which is deliberated to be the consequence of the rapid devolatilization of cellulose and hemicellulose. During this pyrolysis experiment, the temperature is considered as the most significant parameter to determine the product distributions. The heating value of the bio-oil was more than half the value of gasoline and diesel which is good enough for an agricultural waste. The functional group present in the pyrolysis oil is similar to the other bio-oil products. The bio-oil was fractionated into chemical classes by gas chromatography. Through GC-MS, it was found that the bio-oil derived from neem bark was a mixture of various organic compounds of carbons with the chain length in the range of C<sub>5</sub>-C<sub>47</sub> with a lot of oxygenated aromatics (ArO) which is similar to gasoline and diesel fuels. The bio-oil contained significant amounts of complex organic mixtures with phenols, alkanes, alkenes, fatty acids, esters, resulting in low pH values and high oxygen contents which can be used as a feedstock for chemical industries. The physical characteristics of the bio-oil were in the range of moderate quality fuels

**Table 6.** Gas fractions at different operating conditions.

Composition of gas	Temperature °C					Particle size mm				Sweep gas flow rate m <sup>3</sup> /hr			
	350	400	450	500	550	0.6	0.71	1.0	1.25	1.25	1.75	2.0	2.25
Hydrogen (%)	1.62	2.35	4.35	6.22	7.92	0.97	3.33	5.68	4.21	3.45	5.22	7.23	5.21
Methane (%)	0.12	1.25	1.89	1.94	2.26	0.9	1.36	2.22	1.92	3.21	3.01	2.52	2.72
Oxygen (%)	1.21	1.16	0.91	0.88	0.81	0.91	1.11	1.56	1.26	2.45	1.56	1.27	1.24
Carbon dioxide (%)	7.17	7.52	8.11	8.23	6.54	7.56	9.32	12.31	10.34	6.33	8.56	10.96	8.01
Carbon monoxide (%)	2.36	2.82	2.99	3.45	2.07	0.82	1.56	3.21	1.25	1.55	3.42	4.63	3.96
Nitrogen (%)	87.52	84.9	81.75	79.28	80.4	88.84	83.32	75.02	81.02	83.01	78.23	73.39	78.86

and other pyrolysis bio-oils. The pyrolysis gas can be used as a gaseous fuel. The gas fraction is found to have hydrogen, methane, oxygen, with higher rate of carbon dioxide and carbon monoxide. The char produced from this study has an acceptable volatile matter and fixed carbon. The moisture content of the char is minimum when compared with the biomass material which enhances the energy content. Hence, it can be used as an energy source.

## Notes on contributors

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