



Extraction of Bio-Oil From Mahua Seeds and Its Characterization

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Abstract: Bio-oil was extracted from mahua seeds collected from forests of Chhattisgarh. An intermediate pyrolysis method was used for this process. Proximate analysis of sun-dried mahua seeds shows a high content of volatile matter (86%) and a low content of moisture (4.37%) and ash (1.36%). Fixed carbon content was found to be 8.27%. Particle size of biomass affects the yield of bio-oil, biogas, and biochar. Influence of temperature on the end products of pyrolysis was also studied. Thermogravimetric analysis of mahua bio-oil was carried out to determine the active pyrolysis zone. Decomposition of cellulose and hemicellulose, resulting in heavy weight loss in the seeds, occurs in the temperature range of 150 °C to around 500 °C; thus, it is regarded as the zone of active pyrolysis for mahua seeds. The highest yield of bio-oil is achieved at the temperature of 500 °C. The FTIR study indicates the presence of different aliphatic and aromatic compounds in the seeds. The calorific value of mahua oil was calculated to be 21.38 MJ/kg.

Keywords: Bio-oil, biochar, biogas, biofuel, calorific value, FTIR, mahua seeds, TGA.

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1. Introduction

The development of industrial, transformational, agricultural, power generation, and other sectors in any country depends on energy. Fossil fuels are the main source of energy, but they are limited and will not be able to meet rising energy demand in the world in the long run (Varma et al., 2018). Biomass has the potential to be an alternate source of energy. Two common methods used to convert biomass into energy are biochemical conversion and thermochemical conversion. The biochemical method suffers the demerit of being slow and inefficient. The thermochemical technique involves heating biomass in the presence of a catalyst for conversion of biomass into energy. Pyrolysis is a thermochemical technique for conversion of biomass into biochar, bio-oil, and synthesis gas (Tekriwal et al., 2015). Pyrolysis is a better thermochemical technique as compared to combustion and gasification. Pyrolysis can be classified into three categories: slow, fast, and intermediate pyrolysis (Tripathi et al., 2016).

Mahua trees are available in abundance in the forests of India in states like Chhattisgarh, Bihar, Andhra Pradesh, Madhya Pradesh, Orissa, and Uttar Pradesh. Botanical details of mahua are as follows:

Class: Magnoliopsida

Order: Ebenales

Family: Sapotaceae

Genus: *Madhuca*

Species: *indica*

Mahua seeds have nearly 35% oil and 16% protein. Generally, mahua oil is used for lighting lamps in villages, but it has certain medicinal properties too and also has the potential to be used as a biofuel (Ramadan et al., 2006; Pradhan et al., 2016). In this study, oil is extracted from mahua seeds using the intermediate pyrolysis method. Characterization of seeds as well as extracted oil is done to understand their properties.

2. Materials and Methods

Seeds of mahua were collected from the southern parts of Chhattisgarh. Proximate analysis gives information about the moisture content, fixed carbon, volatile matter, and ash content present in the sample. Hence, it is one of the characterizations that needs to be performed initially before using the biomass as a fuel. Yields of bio-oil, biogas, and char obtained by the pyrolysis of biomass of

different sizes are recorded. An electric hot air oven was used for heating 1 g of seeds for 1 hour at 105-110 °C. The sample was cooled gradually by placing it in the desiccator and then reweighed. A reduction in the weight of the sample occurs due to heating. This difference in weight gives information about the initial moisture present in the seeds. The sample collected after desiccation is free from moisture. Thereafter, it is placed in a silica crucible with a lid. A muffle furnace is used to heat the crucible. The temperature is maintained at 925 ± 20 °C for 7 minutes. After heating, the crucible is removed from the furnace and allowed to cool. The sample is then weighed again. The loss in weight indicates the volatile matter content present in the seeds. Next, the sample (without a lid) is placed in an oven and heated at 700 ± 20 °C. This heating process is continued for 30 minutes. The leftover sample represents the ash content of the seed. The content of fixed carbon in seeds is calculated using the following formula.

$$\text{Percentage of fixed carbon} = 100 - \text{Percentage of (ash + moisture + volatile matter)} \quad (1)$$

Mahua seeds were dried in the sun for 5-6 days to remove moisture. The sun-dried seeds were then crushed and placed in an alumina crucible. The crucible was heated to a temperature ranging from 25 °C to 700 °C with a heating rate of 10.0 K/min. Nitrogen gas was purged at a flow rate of 70.0 mL/min to maintain an inert atmospheric condition. This displaces air in the pyrolysis zone, which may result in avoiding unwanted oxidation of the sample.

Following this, the TGA curve is plotted with the weight of crushed seeds (in mg) on the y-axis and temperature (in °C) on the x-axis. The DTG curve is plotted as weight loss per unit temperature versus temperature. For TGA, approximately 4.3450 mg of crushed mahua seeds was taken. TGA and DTG profiles were recorded using a METTLER TOLEDO TGA 1 module.

Lastly, FTIR (Fourier transform infrared) spectroscopy helps in the determination of different functional groups present in the sample. The range of wavenumbers used is 600–4000 cm^{-1} . The FTIR study was done using an ALPHA ATR-ZnSe model (Bruker).

3. Results and Discussion

3.1 Proximate Analysis

Proximate analysis of the sun-dried seeds was done to determine the fixed carbon content, moisture content, volatile matter, and ash content in the seeds. This gives

information about the quality of biofuel. The proximate analysis of mahua seeds is shown in Table 1.

Table 1. Proximate analysis of mahua seeds.

Seed	Moisture (%)	Volatile matter (%)	Ash (%)	Fixed-carbon(%)
Mahua	4.37	86	1.36	8.27

The heating value of biomass drops when it has high moisture content. A small amount of moisture (less than 5%) and an excess of volatile matter (more than 80%), accompanied by fixed carbon content, improve the quality of biomass as they contribute to the high heating value of the bio-residues. Ignition of fuel also becomes easy as the volatile matter is present in large amounts. The fixed carbon content acts as the main heat generator on burning biomass. Ash is obtained as the residue when the biomass is burnt. This ash acts as a sink. It is known that high ash content lowers the calorific value. The ash content is low for mahua seeds (i.e., 1.36%). A similar observation was also made by Pradhan and Singh (2013).

3.2 Effect of Particle Size on Bio-Oil Yield

During pyrolysis, biomass undergoes thermal degradation. It takes place in an oxygen-free environment. The rate at which heat is transferred to the biomass and the yields of the end products depend upon the particle size (Varma et al., 2018). Table 2 shows the variation in the yields of biogas, bio-oil, and char for various biomass particle sizes.

Table 2. Yields for variation in particle size of mahua seed.

Particle size	Bio-oil (%) yield)	Biochar (%) yield)	Biogas (%) yield)
0.15 μ m	40	21	39
0.30 μ m	43	24	33
2 mm	47	26	22
4 mm	46	38	16

Vapors are formed when thermal cracking of biomass takes place. The flow of heat from the hot end to the cold end slows down in bigger particles because the aid biomass and its core become distant. As a result of this, the distance that the vapors travel through the char increases and thus, more char is formed (Tripathi et al., 2016; Park et al., 2012; Guedes et al., 2018). Yields (biogas, bio-oil, and biochar) obtained for various biomass particle sizes are illustrated in Figure 1.

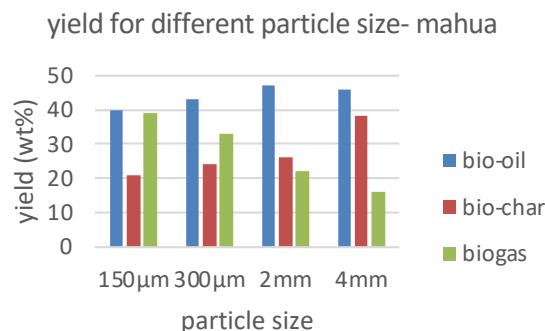


Figure 1. Variation of bio-oil yield for different biomass particle sizes.

3.3 Thermal Degradation Profile for Mahua Seeds

In the pyrolysis of biomass, thermal decomposition of material occurs at high temperatures in an oxygen-free atmosphere. The effective temperature of mahua seeds can be determined by studying the TGA. A graph is plotted to analyze the weight loss (thermogravimetric) in the sample as the temperature increases. The temperature range where the highest thermal degradation takes place can be studied using this graph.

Figure 2 illustrates the TGA curve plotted between weight (in mg) and temperature (in $^{\circ}$ C) and the DTG curve plotted between weight per unit temperature (in mg/ $^{\circ}$ C) and temperature (in $^{\circ}$ C) for mahua seeds. It can be seen that during thermal degradation, approximately 4.58% weight loss occurs up to 170 $^{\circ}$ C as the moisture and other volatile compounds get removed. Due to the low moisture and low volatile content of the seeds, the weight loss at this stage is low (Pradhan & Singh, 2013).

The second stage is the temperature range of 170–530 $^{\circ}$ C, which shows maximum weight loss (nearly 77%). This may be attributed to the decomposition of cellulose and hemicellulose in mahua seeds. This stage can be characterized as the active pyrolytic zone due to high weight loss. The active pyrolytic zone consists of two stages, in which the hemicellulose degrades first, and then cellulose breaks up into lighter components.

In the third stage, weight loss becomes nearly constant after 530 $^{\circ}$ C as the lignin decomposes very slowly from 530 to 700 $^{\circ}$ C. This stage is observed as the passive pyrolysis zone. The slow and continuous weight reduction in this zone may be due to the degradation of complex high-molecular weight such as lignin present in components of mahua seeds due to the thermal environment. The residue left after thermal degradation of mahua seeds is around 0.5889 mg, which is nearly 13.5540%

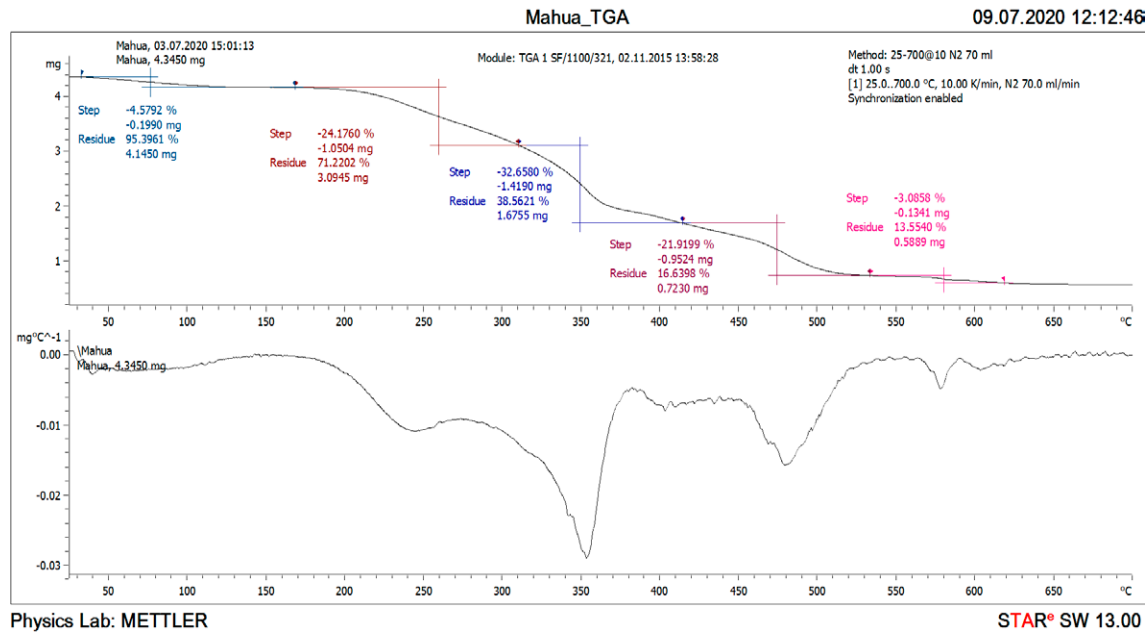


Figure 2. TGA and DTG plot of mahua seeds.

of the sample taken. This residue generally comprises mineral constituents and chemically bound carbon. The presence of lignin results in the production of biochar at the end (Guedes et al., 2018; Protić et al., 2018; Singh et al., 2020; Prasad Shadangi & Mohanty, 2013)

The DTG profile for mahua seeds shows a weight loss per °C as the temperature is increased. The peaks observed around 100-130 °C are a result of loss of moisture and other volatile compounds. The degradation peaks for cellulose and hemicellulose are observed around 350-450 °C, whereas peaks around 500 °C show the degradation of lignin. Lignin contains phenolic hydroxyl group compounds, which increase its thermal stability; hence, lignin degrades at higher temperatures, i.e., around 500 °C or above.

Mahua seeds show lower lignin content, as the amount of char produced as residue at the end is the minimum. Lower lignin content and higher hemicellulose and cellulose content show that the quantity of char formed will be less and the liquid fuel yield will be higher (Protić et al., 2018).

3.4 Variation in Yield for Different Temperatures

Table 3 shows the variation in product yields at different temperature ranges extracted from mahua seeds. With increasing pyrolysis temperature, the yield of biogas increases, but biochar decreases. This indicates that the

process of pyrolysis continued at higher temperature. The highest bio-oil yield was reported at 500 °C. TGA results support the explanation of lower yield after 500 °C. At elevated temperature, the breakdown of cellulose and hemicellulose gets accomplished, and only the lignin is left to decompose, resulting in biochar formation. The yield of bio-oil decreases at higher pyrolysis temperature as the production rates of condensable molecules, which happen due to decomposition of lignin, are less than the rate at which cracking of condensable molecules into non-condensable gases takes place (Tripathi et al., 2016; Pradhan & Singh, 2013; Singh et al., 2020). Figure 3 illustrates the variation in product yield for different temperatures.

Table 3. Bio-oil yields extracted at different temperature ranges for mahua seed.

Temperature (°C)	biooil (%yield)	biochar (%yield)	biogas (%yield)
450	37	29	18
475	40	28	21
500	47	26	22
525	39	21	23
550	36	20	26
575	29	18	29

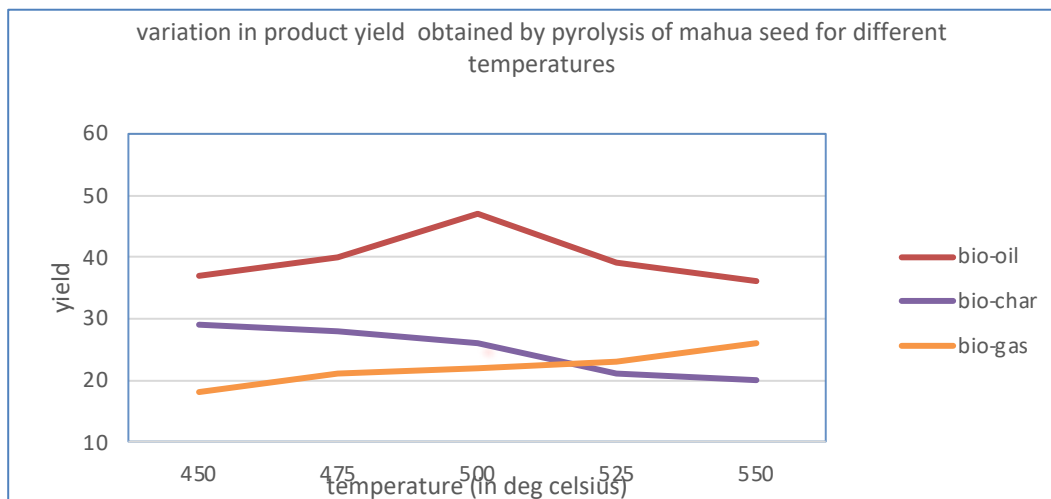


Figure 3. Variation in product yield for different temperatures.

3.5. FTIR Study of Mahua Seeds

Fourier transform infrared spectroscopy (FTIR) study is the most commonly used technique to identify the functional groups that constitute the sample. Figure 4 illustrates the FTIR spectrum of mahua seeds. A broad peak from 3500 cm^{-1} to 3200 cm^{-1} can be seen in the spectrum, which represents the presence of moisture, carboxylic acid, and phenols in the mahua seeds. It is due to O-H stretching. It shows that fiber is present in the seeds. The presence of alkanes is shown by the absorption peak at $2950\text{--}2851\text{ cm}^{-1}$ and can be attributed to the axial deformation of the C-H group. A major peak can also be observed at 1744.17 cm^{-1} that supports the presence of a carbonyl group, such as a ketone. This shows that hemicellulose is present in the seeds. A C=C stretching vibration can be seen in the range of $1600\text{--}1500\text{ cm}^{-1}$ that proves the presence of alkenes and aromatic compounds. The range $1470\text{--}1300\text{ cm}^{-1}$ contains peaks that may be due to C≡C stretching, which shows the presence of alkynes. The presence of ester and ether in the seeds is shown by the peaks in the $1280\text{--}990\text{ cm}^{-1}$ range and can be attributed to C-O stretching. A symmetric deformation of C-O-C of the cellulose and hemicellulose may have resulted in the peak at 1159.82 cm^{-1} . An absorption peak is observed in the range $800\text{--}650\text{ cm}^{-1}$. This may be because of O-H bending and supports the presence of mono- and polycyclic substituted aromatic groups. The study shows the presence of a large number of aliphatic groups along with a smaller number of aromatic groups in the seeds.

This explains the presence of cellulose, hemicellulose, and lignin in mahua seeds (Pradhan & Singh, 2013; Prasad Shadangi & Mohanty, 2013).

3.6. Calorific Value

A bomb calorimeter was used to study the calorific value of mahua biochar. The calorific value of biochar obtained from mahua seeds was calculated to be 21.38 MJ/kg .

4. Conclusions

The present study discusses the extraction of bio-oil from mahua seeds from forests of Chhattisgarh and its characterization using intermediate pyrolysis, proximate analysis, FTIR spectroscopy, and a bomb calorimeter. Proximate analysis shows low moisture content, low ash content, and high content of volatile matter present in mahua seeds. In the initial stage of thermal degradation, volatile components and moisture are removed, which results in weight loss. In the pyrolysis zone, the breaking up of cellulose and hemicellulose into water, CO, CO₂, and some organic compounds of small length as main products takes place. The degradation of hemicellulose takes place at a lower temperature as compared to cellulose. In the passive pyrolysis zone, lignin degrades. It occurs at high temperature because of the presence of phenolic hydroxyl groups, which increases its thermal stability. Lignin breaks up into aromatic compounds, CO and CH₄ gases, and small-chain organic compounds. Char is also obtained as a by-product which is obtained because of lignin present in biomass. High moisture and high lignin content in the material reduce the combustion efficiency. Lignin content and moisture are found to be very low in all three seeds. The FTIR study shows the presence of carbonyl groups along with aliphatic and aromatic compounds in all three seeds, which provides evidence for

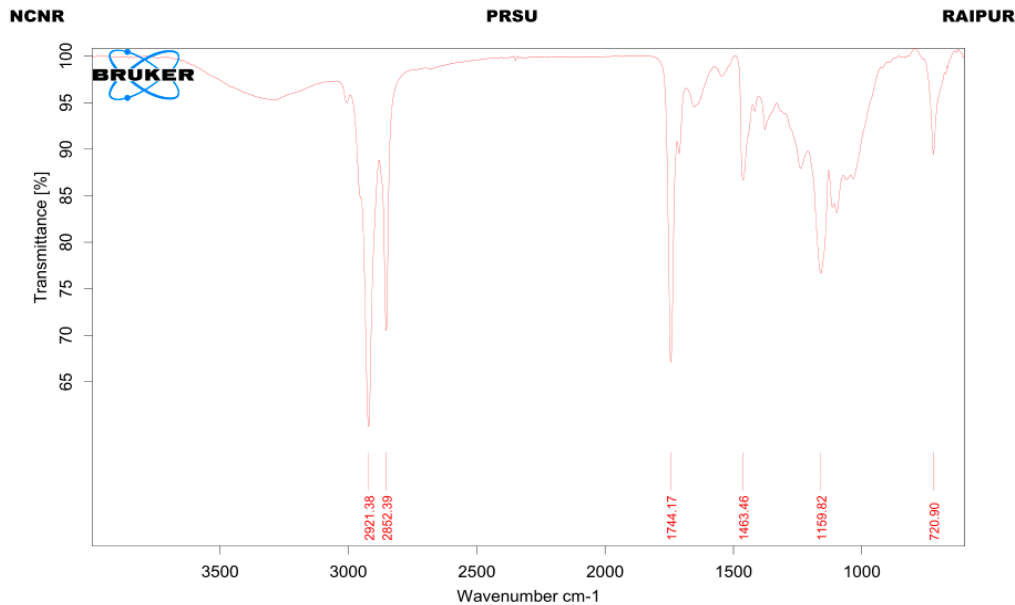


Figure 4. FTIR spectrum of mahua seeds.

the presence of hemicellulose, cellulose, and lignin in the seeds. This study shows that mahua seed has the potential to be used in the pyrolysis process for the production of biofuels.

5. Future Scope

The current method provides a reliable approach to estimating the volatile content in seeds through thermal treatment and weight loss analysis. In the future, this process can be further optimized by integrating automated temperature control systems and real-time weight monitoring to improve accuracy and efficiency. Additionally, exploring the use of advanced analytical tools such as thermogravimetric analysis (TGA) or gas chromatography could offer more detailed insights into the composition and behavior of volatile compounds. The methodology can also be adapted for a wider variety of biological samples, enabling its application in seed quality assessment, food processing, and agricultural research.

Conflict of Interest

The authors have no conflict of interest to declare.

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