



# Investigation Of Process Parameters for Producing Bio-Oil from Luffa Cylindrical Fiber in a Fixed Bed Reactor Using Pyrolysis Process

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## ABSTRACT

*This study was performed to assess the impact of pyrolysis parameters on the yield of pyrolytic bio-oil during the thermal degradation of luffa cylindrical fiber in a fixed-bed reactor. The study revealed that the optimal bio-oil output of 29 wt% was attained at a reactor temperature of 600 °C, a biomass particle size of 4 mm, and a nitrogen gas flow rate of 1.5 L/min. The Gas Chromatography-Mass Spectrometry (GC-MS) analysis of the bio-oil revealed the presence of phenols, alcohols, carboxylic acids, ketones, alkenes, alkanes, aldehydes, and aromatics, indicating that the pyrolysis of luffa cylindrical fiber could be a viable approach for producing renewable fuels and chemicals while mitigating environmental pollution concerns.*

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## INTRODUCTION

Energy catalyzes the advancement of the global economy. Fossil fuels, including petroleum, coal, and natural gas, fulfill the predominant portion of global energy requirements (Anas *et al.*, 2024). The combustion of fossil fuels generates greenhouse gas emissions, which adversely impact the environment. Furthermore, there are constraints on the accumulation of fossil fuels. The energy demand is increasing due to development (Biswas *et al.*, 2017). In response to the increasing energy demand and environmental challenges, the global trend is shifting towards sustainable energy production, diminished vehicular and industrial emissions, greenhouse gas reduction, waste minimization, and decentralized

electricity generation (Iwekumo and Samuel, 2019). Renewable biomass energy contributes to addressing each of these energy and environmental issues (Iwekumo and Samuel, 2019).

Thermochemical techniques, including pyrolysis, enable the liquefaction of biomass and can substantially enhance the petrochemical industry and the shift towards bio-based refineries (Iwekumo and Samuel, 2019). Fluids made from renewable materials can be used in the petrochemical industry in different ways: (1) blended with fossil fuels as a drop-in fuel, (2) as a complete substitute for fossil fuels, or (3) as raw materials in new conversion methods. Encouraging the use of biobased feedstocks

in the petrochemical industry facilitates the maintenance of essential elements of the existing infrastructure for the production, distribution, and consumption of chemicals and transportation fuels while reducing global reliance on fossil feedstocks (Balogun *et al.*, 2019). However, liquids made from biomass through pyrolysis have complex and problematic characteristics that make them difficult to use directly in existing systems. Liquids made from biomass through pyrolysis are not stable when heated and have high acidity, mainly because they contain a lot of oxygen and a wide range of different chemicals that can react in various ways. Consequently, the biomass-to-liquid process requires additional advancement, such as improving the selectivity of specific chemicals. This process development may encompass biomass pre-treatment, optimization of the biomass devolatilization phase, and subsequent vapor reforming (Zhang *et al.*, 2021).

Biomass is a complex, heterogeneous substance that necessitates additional research to fully realize its potential (Kumar *et al.*, 2019). Examples of significant biomass resources include woody biomass, energy crops, equine manure, and agricultural waste (Onokwai *et al.*, 2022b). Cellulose, hemicellulose, and lignin produce a variety of compounds in the resulting liquids, which can be utilized for chemical synthesis and transportation fuels. (Go *et al.*, 2019). Consequently, meticulous process development for biomass-to-liquid conversion, informed by biomass chemistry and industrial requirements, is of paramount importance (Go *et al.*, 2019). The transformation of biomass into electricity has garnered heightened interest recently. Biomass is a renewable, low-sulfur energy source characterized by a balanced carbon cycle (Mohapatra and Gadgil, 2013). Consequently, biomass energy is

poised to emerge as a significant energy source in the future, particularly in tropical nations (EU Commission, 2018). So, a lot of focus has recently been put on finding the right types of biomass that can produce a lot of energy to replace traditional fossil fuels.

The research conducted by Gao *et al.* (2020) demonstrates that *Luffa cylindrica* fiber possesses favorable attributes for pyrolysis, such as a substantial cellulose content and a minimal ash content. These properties facilitate the production of bio-oil with a reduced oxygen level and biochar with elevated carbon content. The study highlights the possibility of *Luffa cylindrica* fiber as a sustainable raw material for pyrolysis, underscoring its adaptability in the applications of bioenergy generation and environmental remediation. A study by Abbas *et al.* (2018) looked at how rice husk ash (RHA) breaks down in pyrolysis and found that the best amounts of biochar and bio-oil produced were 39% and 19%, respectively, when heated to 500 °C. A significant decrease in acidic value and carboxyl groups was seen at elevated temperatures of 700 °C, although biochar generated at higher temperatures exhibited remarkable stability. The final product comprises liquid chemical components ranging from 22.5% to 31.7%, gaseous compounds ranging from 27.7% to 42.5%, and solids ranging from 34.1% to 42.5%. Gui *et al.* (2014) assessed the efficiency of fast and slow pyrolyzed bio-oil production methods, specifically examining the control variables of temperature, heating rate, and yield. The study evaluated the composition of coconut shells, RHA, and a mixture of 50% of each component to set the standards. Islam *et al.* (2017) evaluated the optimal yield coefficients for bio-oil production from biomass solid waste in a fixed bed reactor maintained at 500

°C. An analysis of the product reveals that 30% of liquid goods and 33% of solid products are produced by the pyrolysis of solid waste. Hu *et al.* (2015) performed an experimental investigation on the pyrolysis of water hyacinth to produce syngas in a quartz tube reactor, analyzing the effects of different particle sizes, temperatures, and catalysts to identify optimal conditions for syngas generation. Biswas *et al.* (2017) performed a comparative study on the effect of temperature on bio-oil yield during the slow pyrolysis of azolla, sargassum tenerrimum, and water hyacinth. The highest liquid product yields were 38.5 wt% for Azolla, 43.4 wt% for Sargassum tenerrimum, and 24.6 wt% for water hyacinth.

There isn't much research on how the size of biomass particles and the flow rate of inert gas affect the production of pyrolytic bio-oil from *Luffa cylindrica* fiber. Understanding how pyrolysis conditions affect the types of products produced is important for improving and controlling the process; therefore, this study aims to thoroughly examine how pyrolysis conditions (especially temperature, biomass particle size, and inert gas flow rate) affect the behavior of *Luffa cylindrica* fiber in producing pyrolytic bio-oil in a fixed bed reactor.

## MATERIALS AND METHODS

### Collection of *luffa cylindrica* fibre biomass samples

The *luffa cylindrica* fibre used in this study was obtained from farmland in Amawom village in Ikwuano local government area of Abia state, Nigeria which lies within latitude 5°20' to 5°32' N and longitudes 7°32' to 7°40' E. All foreign materials were removed by washing to ensure the sample was free from impurities.

### Preparation of sample

The samples of *luffa cylindrica* fibre were sun dried for ten days to a constant weight and then ground in a high-speed rotary cutting mill. The ground *luffa cylindrica* fibre samples were separated into various size distributions using a standard sieve into particle size 1mm, 3mm, 4mm, 5mm, 6mm, and 7mm and were stored or collected in different desiccators until needed.

### *luffa cylindrica* fibre sample analysis

#### Proximate analysis of *luffa cylindrica* fibre

Proximate analysis quantifies the percentages of volatile matter, ash, moisture content, and fixed carbon in a biomass sample. The methodology for estimating each of these values adheres to ASTM standards as referenced in the literature (Marquez-Montesino *et al.*, 2015; Wauton and WilliamEbi, 2019).

#### Ultimate Analysis of the *luffa cylindrica* fibre

This test aims to ascertain the elemental percentages of carbon, hydrogen, nitrogen, sulfur, and oxygen in *luffa cylindrica* fibre. The final analysis was performed by ASTM standards E777 and E778.

#### TGA (Thermal gravimetric analysis) of *luffa cylindrica* fibre

Thermo-analytical measurements were conducted using a Mettler-Toledo TG850 thermo-analyzer, which simultaneously recorded Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) curves. The analysis was performed under a high-purity, dried nitrogen atmosphere with a constant flow rate of 0.08 L/min. A continuous evacuation was maintained at a pressure of  $1.33 \times 10^{-6}$  mbar throughout the experiment.

The temperature range for the analysis was from room temperature to 1000 °C, with a consistent heating rate of 10 °C/min. Alumina (Al<sub>2</sub>O<sub>3</sub>) was used as the reference material for DTA measurements.

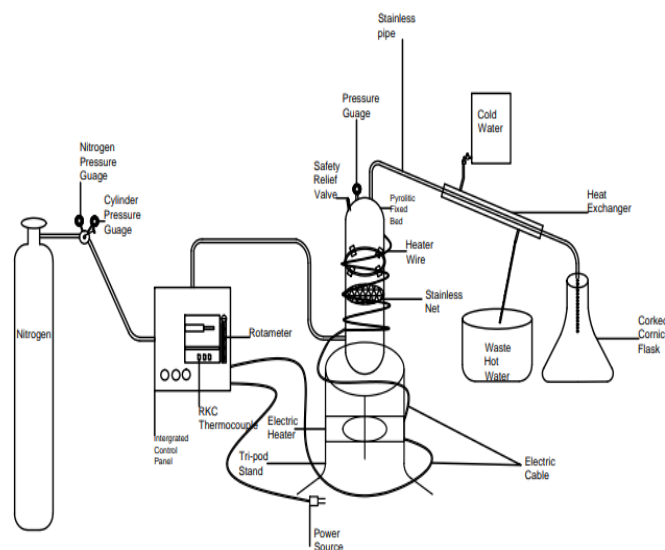
### Experimental set-up

The experimental equipment has been built to facilitate experimental tests under effective heat control of the processed sample while enabling the processing of a sufficient sample quantity to collect liquid products for offline analysis. Pyrolytic bio-oil is often corrosive, and the operational temperature range is 200–700 °C (Uddin *et al.*, 2021). The primary components of the system were constructed from SUS 304-grade stainless steel. The entire fabrication process occurred at Dyke’s Fabrication Workshop in Ihie Ndume, Umuahia, Abia State, Nigeria.

The reactor comprises a stainless-steel tube with a height of 300 mm, an internal diameter of 94 mm, and an external diameter of 100 mm. The reactor's modest diameter permits the disregard of the temperature gradient within the feed bed (Borello *et al.*, 2014), and a proportional integral derivative (PID) temperature controller (Berme, model: REX-C100FK02-VAN DN) ensures a uniform temperature in the reactor; consequently, the entire process is considered isothermal. The upper portion of the reactor permits the entrance of raw material (biomass), and char can be recovered after the experiment's conclusion. The reactor is heated by two devices: a hot plate and vertically wound electrical heating coils. The temperature of the reactor is gauged with a K-type thermocouple. The pyrolyzer is insulated with fiberglass on the exterior to minimize heat loss to the environment. Nitrogen gas was heated at the reactor's surface before being introduced into

the reaction system to avert a reduction in reaction temperature. An exit pipe at the reactor's apex conveys the gases produced during pyrolysis to the condenser, which is tilted at roughly 15 degrees to promote liquid product drainage and prevent accumulation (Chaiya and Reubroycharoen, 2013). The condensed liquid, referred to as pyrolytic bio-oil, is gathered in a beaker.

A small porous plate or mesh is situated at the reactor's base to support bed materials and facilitate uniform dispersion of N<sub>2</sub>, so preserving an inert atmosphere throughout the experiment. A nitrogen gas flow meter with a variable control valve capacity of 2.5 L/min was utilized to manage and measure the inert gas flow rate. Nitrogen gas replaces air in the reactor to create an anoxic environment and functions as a carrier or sweeping gas for the pyrolysis gases. Figure 1 illustrates a schematic depiction of the experimental setup utilized for the fixed bed pyrolysis procedure.



**Figure 1.** Schematic diagram of the fixed bed pyrolysis experimental system.

### Experimental procedure

### Effect of temperature

Forty grams of dried luffa cylindrical fibre, with an average diameter of 1.0 mm, were injected into the reactor. After establishing all connections, a nitrogen gas flow rate of 1.2 L/min was maintained and measured using a rotameter. The biomass was subjected to heating at temperatures of 550, 600, 650, 700, and 750 °C, maintaining a uniform heating rate of 50 °C/min. The liquid oil and the solid residue that remained in the reactor tube were weighed post-pyrolysis. The gas produced was determined using material balancing. The temperature associated with the maximum liquid yield in the initial run was designated as a constant parameter for the second run. Three trials were performed, and the mean was computed.

### Effect of particle size

In the second trial, five distinct particle size ranges of 3mm, 4mm, 5mm, 6mm, and 7mm were selected as the variable parameter to assess the impact of particle size on bio-oil yield, utilizing the highest liquid yield temperature established in the initial trial. Triplicate trials were conducted, and the average was calculated.

### Effect of inert gas flow rate

The flow rate of the inert gas is directly correlated with the vapor residence time. The impact on bio-oil yield was examined by varying the maximum yield parameters for temperature, particle size, and carrier gas flow rate at 0.5, 1.0, 1.5, 2.0, and 2.5 L/min, with the highest yield identified. Triplicate trials were performed, and the average was calculated.

### Bio-oil composition

Gas chromatography-mass spectrometry analysis was conducted using a Perkin Elmer Turbo Mass Spectrophotometer (Norwalk, CTO6859, USA),

equipped with a Perkin Elmer Auto Sampler XLGC. The employed column was a Perkin Elmer Elite -5 capillary column, measuring 30m × 0.25mm, with a film thickness of 0.25mm, composed of 95% Dimethylpolysiloxane. The carrier gas utilized was helium at a flow rate of 0.5 mL/min. A sample injection volume of 1 µl was utilized. The inlet temperature was maintained at 250°C. The oven temperature was first set to 110°C for 4 minutes, then increased to 240°C. Subsequently, increase the temperature to 280°C at a pace of 20°C per minute, maintaining this for 5 minutes.

The overall time was 90 minutes. The mass spectrometry (MS) transfer line was maintained at a temperature of 200°C. The source temperature was maintained at 180°C. GCMS was examined by electron impact ionization at 70 eV, and the data was evaluated through total ion count (TIC) for compound identification and quantification. The spectra of the components were juxtaposed with the database of established component spectra cataloged in the GC-MS library. Peak area measurements and data processing were performed utilizing Turbo-Mass OCPTVS-Demo SPL software.

## DISCUSSION

### Results of characterization Of *Luffa Cylindrica* fibre

#### Proximate analysis result

Table 1 displays the proximate analysis results for the *luffa cylindrical* fibre sample. The values for ash content, moisture content, volatile matter, and fixed carbon in the sample were 18.25, 7.60, 43.40, and 30.75 (% w/w), respectively, while the calorific value was 20.62 kJ/g. The *luffa cylindrical* fibre proximate analysis results showed that it contained more volatile matter and fixed carbon. These components

are important for oil production (Siqueira *et al.*, 2008). The presence of low concentrations of moisture content and ash content favors the pyrolysis process considerably. This result is in close agreement with Pedro *et al.* (2022); and Anas *et al.* (2024).

**Ultimate Analysis Result**

The ultimate analysis assessed the elemental compositions of the samples. As shown in Table 2 below, the values for carbon, hydrogen, nitrogen, oxygen, and sulfur in the sample were 65.70, 5.98, 1.12, 10.80, and 0.80 mass%, respectively. The elevated carbon and oxygen content signifies that luffa cylindrical fibre consists of highly polar structures, advantageous for the mechanism of ion exchange adsorption.

**Table 1: Proximate Analysis of the *luffa cylindrica* fibre**

Item	% value
Ash content	18.25 ± 0.2
Moisture content	7.60 ± 0.4
Cal Value (Kj/g)	20.62
Volatile content	43.40 ± 0.7
Fixed carbon content	30.75 ± 0.4

**Table 2: Ultimate Analysis of *Luffa Cylindrical Fibre***

Elements	Wt %
Carbon	65.70 ± 0.3
Hydrogen	5.98 ± 0.3
Nitrogen	1.12 ± 0.1

Oxygen	10.80 ± 0.2
Sulfur	0.80 ± 0.3

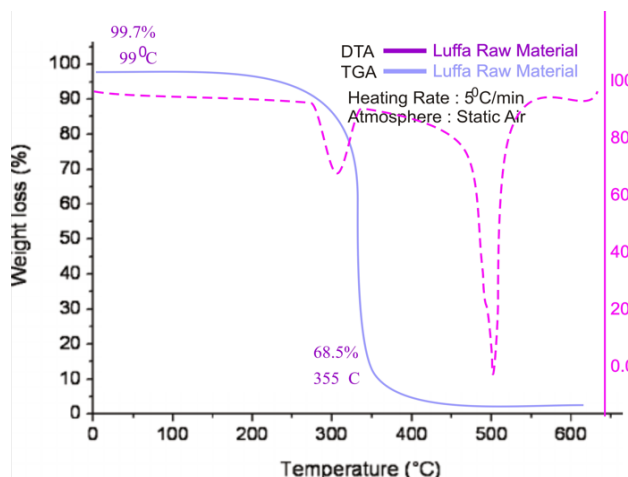
Similarly, the low sulfur and nitrogen content may mitigate the accumulation of deleterious chemicals in the reactor compartment, thereby decreasing the maintenance requirements of the pyrolysis plant. The reduced sulfur and nitrogen levels may result in a decrease in SO<sub>x</sub> and NO<sub>x</sub> emissions, which negatively impact the ozone layer in the troposphere (Ayeni *et al.*, 2018). The results compared well with Anas *et al.* (2024) and Narayan *et al.* (2021).

**TGA/DTA (Thermal gravimetric analysis) results of *luffa cylindrical fibre*.**

Figure 2 shows the thermogravimetric profile that outlines how luffa cylindrical fiber breaks down when heated at a rate of 5°C per minute, showing weight loss related to temperature. The profile indicates that most weight loss occurs during the temperature range of around 250 to 400 °C. The initial weight loss is attributed to the removal of moisture and light volatiles from the biomass, followed by the decomposition of volatile substances and the release of volatiles (i.e., devolatilization) occurring between 250 and 400 °C, as indicated by a significant endothermic peak in the TGA.

The volatile matter consists of cellulose and hemicellulose (Nagaraja Ganesh and Muralikannan, 2016). The parts mainly include alkanes, aromatics, esters, alcohols, and ketones, as well as different structures that contain oxygen. This outlines the temperature range for the pyrolysis experiment. The predominant weight loss transpires during this phase. Secondary reactions such as cracking, partial oxidation, polymerization, and repolymerisation

follow devolatilization (Jones, 2011; Amini et al., 2019). This outcome aligns with the findings of Khadir et al. (2020), Valcineide et al. (2014), and Pedro et al. (2022).



**Figure 2:** Thermogram curve of raw *Luffa cylindrical* fibers.

### Effects of operational variables on bio-oil yield

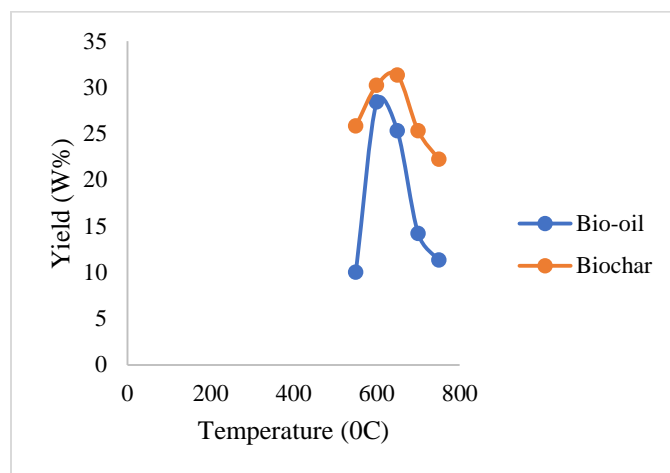
#### The effect of pyrolysis temperature on bio-oil yield

Temperature is one of the most important factors in pyrolysis because it affects the feed material's thermal degradation. We kept other parameters constant in this study at 1mm for particle size and 1.2 L/min for inert gas flow rate, respectively. The required experiments were done at various temperatures of 550, 600, 650, 700, and 750 °C to determine the different yields of bio-oil and biochar. The maximum bio-oil and biochar yield of 29% and 31% occurred at temperatures of 600°C and 650°C, respectively. There was a clear observation that, by increasing the temperature, the devolatilization of the biomass increased, resulting in increased bio-oil and biochar yield. An increase in the temperature above 650°C resulted in a significant decrease in the yield of the pyrolysis product. The reason may be that there has been a complete devolution of the feed

material, and secondary cracking started taking place. The findings compared well with Sukumar *et al.* (2015) and Kim *et al.* (2010).

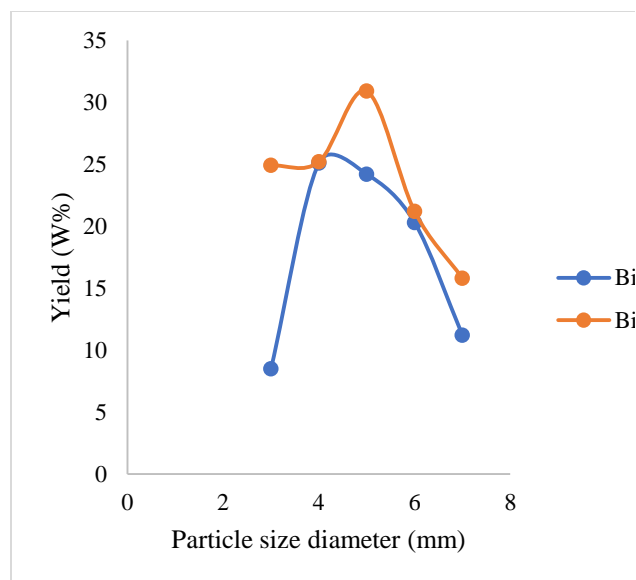
#### The effect of particle size on bio-oil yield

The particle size diameter was varied from 3mm to 7mm to determine its effect on the yield of pyrolysis



**Figure 3:** Effect of temperature on the pyrolysis process

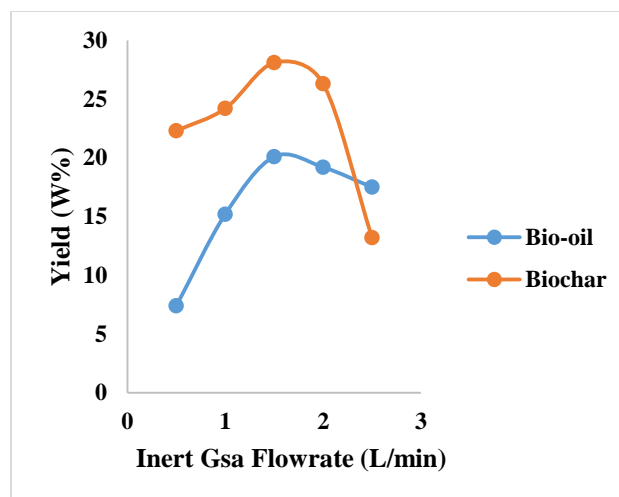
product utilizing a temperature and gas flow rate of 600°C and 1.5 L/min. The percentage yield increased steadily from 3mm to 4mm for bio-oil and 3mm to 5mm for biochar. Beyond 4mm for bio-oil and 5mm for biochar, the yield starts decreasing, as seen in Figure 4. The decrease may be due to a lack of enough surface area for the thermal degradation to take place. This result is in line with Uzun et al. (2006) and Kim et al. (2010).



**Figure 4:** Effect of particle size diameter on the pyrolysis process

#### Effect of inert gas flow rate on bio-oil yield.

The effect of inert gas flow rate on the yield of pyrolysis of *luffa cylindrical* fibre is shown in Figure 5. The percentage yield of the pyrolysis process was examined in groups using inert gas flow rates of 0.5, 1.0, 1.5, 2.0, and 2.5 L/min while maintaining the temperature at 600°C and the particle size at 4mm. The percentage yield increased rapidly with an increase in inert gas flow up to 20.1% and 28.1% at 1.5 L/min for bio-oil and biochar, respectively. Above 1.5 L/min, there was no significant increase in the yield of bio-oil, and there was a significant decrease in the yield of biochar. The insignificant increase in bio-oil yield above 1.5 L/min may be due to complete devolution, while the significant decrease in biochar yield at flow rates above 1.5 L/min may be because of rapid heating, which is known to favor the production of more gas. This result is in agreement with Hossain *et al.* (2014).



**Figure 5:** Effect of inert gas flow rate on the pyrolysis process

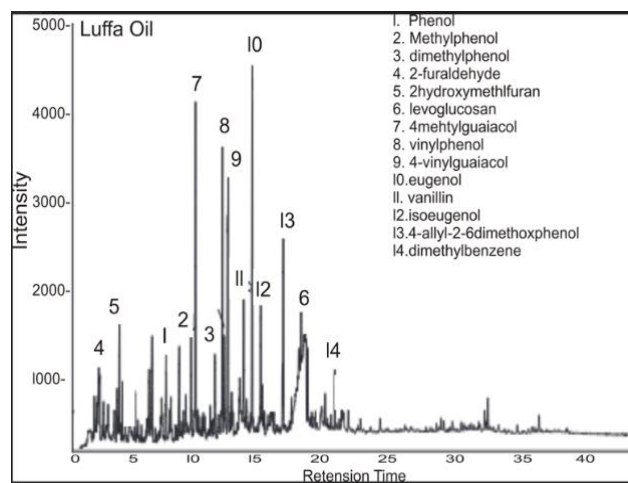
#### Gas chromatography and mass spectrometry (GC-MS) analysis for the bio-oil

Figure 6 presents the bio-oil chemical composition achieved using GC-MS. The compositions of the pyrolytic bio-oil were evaluated considering the relative peak area of identifying constituents using the obtained chromatogram from GC-MS analysis, which agrees with that presented by Laouge *et al.* (2020). The bio-oil is made up of phenol, methyl phenol, dimethylphenol, 2-furaldehyde, 2-hydroxymethyl furan, levoglucosan, 4-methyl guaiacol, vinyl phenol, 4-vinyl guaiacol, eugenol, vanillin, isoeugenol, 4-allyl-2,6-dimethoxyphenol and dimethylbenzene. The most significant components in the bio-oil are 4-methylguaiacol, vinylphenol, 4-vinylguaiacol, eugenol, and 4-allyl-2,6-dimethoxyphenol.

The presence of phenol in the bio-oil was confirmed using FTIR analysis. Levoglucosan is the main product made from cellulose during pyrolysis, which happens through transglycosylation processes. Levoglucosan is very sought after because it has a stable structure, and a specific arrangement of atoms,



and can easily react with other chemicals. Interest in levoglucosan has increased significantly recently owing to its use as a substrate for the production of bio-renewable chemicals in the pharmaceutical and polymer sectors (Nallar and Wong, 2019).



**Figure 6:** GC-MS of bio-oil compound produced from *luffa cylindrical* fibre.

Phenolic compounds, including 4-methyl guaiacol, 4-vinyl guaiacol, and vanillin, are derivatives of lignin (Carpenter *et al.*, 2014). This outcome aligns favorably with the findings of Okokpujie *et al.* (2023), Álvarez-Chávez *et al.* (2019), and Ude *et al.* (2023).

## CONCLUSION

An examination of the influence of pyrolysis parameters on the yield of pyrolytic bio-oil derived from luffa cylindrical fiber in a fixed bed reactor was conducted, leading to the conclusion that the yield of pyrolytic bio-oil increases with temperature, peaking at 600 °C, followed by a gradual decrease. A comparable observation was noted between the biomass particle size and the flow of inert gas. The optimal bio-oil yield was achieved with a particle size of 4 mm and an inert gas flow rate of 1.5 L/min. A maximum bio-oil output of 29 wt% was achieved at a reactor temperature of 600 °C, a biomass particle size of 4 mm, and a nitrogen gas flow rate of 1.5 L/min. Temperature is the predominant factor influencing the pyrolysis behavior of *Luffa cylindrica* fiber in the production of pyrolytic bio-oil. The compositional examination of the bio-oil indicates that it mostly comprises phenols, alcohols, carboxylic acids, ketones, alkenes, alkanes, aldehydes, and aromatics, suggesting its potential for refinement into significant chemicals and fuels.

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