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# Enhanced Bio-Oil Production from Agricultural Waste via $NiCl_2$-Assisted Pyrolysis: A Comparative Study of Candlenut and Coffee Shells

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## Abstract

Indonesia's limited fossil fuel reserves, coupled with increasing national energy demand, highlight the need for alternative and renewable fuel sources. Biomass-derived bio-oil produced through pyrolysis represents a promising solution that can both reduce dependence on petroleum-based fuels and mitigate environmental pollution from underutilized biomass waste. This study investigates the production of bio-oil from candlenut shells and coffee shells through pyrolysis at varying temperatures (250, 350, and 450°C), conducted with and without a $NiCl_2$ catalyst. The bio-oil was characterized for yield, density, and viscosity. The highest bio-oil yields from non-catalytic pyrolysis were achieved at 450°C, amounting to 39.14% for candlenut shells and 41.80% for coffee shells. Catalytic pyrolysis using $NiCl_2$ enhanced the bio-oil yield, producing up to 55.78% (candlenut shells at 450°C) and 58.05% (coffee shells at 350°C). Density measurements showed the highest values in catalytic pyrolysis at 250°C, while the lowest densities were observed in non-catalytic pyrolysis at 450°C. Viscosity followed a similar trend, decreasing with increasing temperature and the presence of the catalyst. FTIR analysis confirmed the presence of functional groups including C–O, C=O, C=C, C≡C, C–H, and O–H. Overall, this study demonstrates the potential of candlenut and coffee shell waste as feasible feedstocks for bio-oil production, offering alternative renewable energy sources for future applications.

## 1. Introduction

The biomass potential in Indonesia is estimated to be 146.7 million tons annually. In the year 2020, the potential for biomass derived from waste amounted to 53.7 million tons [1]. One potential source of biomass energy in Indonesia is waste from agriculture, plantations, and forestry that is currently underutilized. Biomass waste containing lignocellulose can be converted into an alternative energy source through methods such as pyrolysis. Lignocellulose is the primary constituent in the production of bio-oil generated via the pyrolysis process. Pyrolysis is a thermochemical decomposition that occurs in the absence of oxygen, yielding products in three phases: solid, liquid, and gas. The liquid product is referred to as bio-oil, the solid product is known as char, and the gaseous product comprises non-condensable substances. Bio-oil, the liquid product of biomass pyrolysis, is a promising alternative energy source that can replace fuel oil or diesel [2].

Several biomass wastes that are potential feedstocks for bio-oil production include candlenut shells and coffee husks. Candlenut shells have a composition of 48.47% hemicellulose, 27.14% cellulose, and 13.79% lignin; therefore, they have significant potential for bio-oil production [3]. This is supported by the considerably high production of candlenuts in Indonesia, which reached 1,722 tons per year (Statistics Indonesia/BPS, 2018). Processing each kilogram of candlenuts yields 30% kernels and 70% shells. Candlenut kernels are widely utilized as a culinary spice and in various industrial products. In contrast, the candlenut shells are generally not yet optimally utilized or managed to increase their commercial value [4].

Consequently, these shells typically become waste, and without a proper solution, the volume of this waste will continue to increase. Therefore, candlenut shell waste can be utilized as an economically valuable feedstock for bio-oil production. Coffee husks are a form of biomass waste with a high lignocellulosic content, comprising 24.5% hemicellulose, 34.2% cellulose, and 23.4% lignin, indicating their potential for bio-oil production [5]. Coffee husk waste is abundant due to the high productivity of coffee cultivation in Indonesia.

According to Indonesia's Central Bureau of Statistics (2022), coffee production increased to 794.8 thousand tons, a 1.1% rise from the 786.057 thousand tons produced in 2021. The processing of coffee yields approximately 35% coffee husks and 65% coffee beans by weight [6]. Waste in the form of candlenut shells and coffee husks has often been discarded without proper utilization. Therefore, the valorization of these wastes is highly necessary, not only as a potential solution for critical land rehabilitation but also as an alternative fuel source that can contribute to renewable energy development.

Increasing bio-oil products from pyrolysis can be done by adding catalysts. Catalysts function to accelerate the reaction and increase the yield of bio-oil products. The addition of a catalyst to the biomass degradation process can reduce mass loss more effectively than without one, thereby accelerating the pyrolysis process [7]. Catalytic pyrolysis can be carried out with a wide range of catalysts, including acid–base, metal-based, and zeolite catalysts. Among these, metal catalysts such as nickel chloride ($NiCl_2$) are frequently employed due to their ability to enhance deoxygenation, improve bio-oil yield, and promote the formation of valuable hydrocarbon compounds [8]. The nickel catalyst provides high conversion (>60%) and selectivity of 75-95% towards bio-oil. More importantly, the reaction temperature was reduced from around 380°C to 200°C. Nickel is a good catalyst component because it can interact with Lewis acid sites [9].

The pyrolysis of oil palm fiber was conducted at a catalyst-to-fiber ratio of 1:2, utilizing $Al_2O_3$, Al white (white alumina), and $NiCl_2$ as catalysts at a reaction temperature of 400°C. The experimental results demonstrated that among the catalysts tested, $NiCl_2$ exhibited the highest catalytic activity, producing the highest bio-oil yield. Furthermore, the density of the bio-oil obtained from $NiCl_2$-catalyzed pyrolysis was found to be closely comparable to that of conventional biodiesel, indicating its potential as a promising renewable fuel alternative [10]. Catalytic pyrolysis is an effective thermochemical route for converting biomass into biofuels and value-added products with higher efficiency than non-catalytic processes. However, conventional biomass pyrolysis is often energy-intensive and produces oxygen-rich bio-oil with limited fuel quality. Transition metal chloride catalysts, particularly $NiCl_2$, have shown strong potential to overcome these limitations. The presence of $NiCl_2$ can reduce the activation energy of biomass thermolysis by 20–30 kJ/mol and promote the formation of light hydrocarbons ($C_1$–$C_4$). The catalytic activity of $Ni^{2+}$ Lewis acid sites facilitates C-C and C-O bond cleavage in lignocellulosic biomass, resulting in improved conversion efficiency and enhanced product quality [10].

To date, comparative studies of the catalytic pyrolysis behavior of different biomass feedstocks using $NiCl_2$ as a catalyst remain scarce. In particular, there is a lack of systematic comparison between candlenut shells and coffee shells, despite their similar lignocellulosic nature and wide availability in agricultural regions. Understanding how these two biomass types respond to $NiCl_2$-assisted pyrolysis is essential for optimizing bio-oil production and tailoring catalyst selection. This study aims to (1) compare the pyrolysis behavior of candlenut and coffee shells, (2) evaluate the effect of $NiCl_2$ catalyst on bio-oil yield and quality, and (3) characterize the chemical and physical properties of the resulting bio-oils.

## 2. Experimental

### 2.1. Materials

The primary materials used in this study were candlenut shells and coffee husks collected from Pringsewu Regency, Lampung Province. The catalyst employed in this study was $NiCl_2$ with a purity of 99%.

### 2.2. Procedures

This study employed a Completely Randomized Factorial Design (CRFD) with three factors. The pyrolysis process was conducted in a 4-liter batch reactor equipped with an automatic temperature controller and an electric heater. The reactor scheme is shown in Figure 1. The independent variables included the type of feedstock (candlenut shells and coffee husks), the operating temperatures (250°C, 350°C, and 450°C), and the use of a catalyst (none and with $NiCl_2$). The $NiCl_2$ catalyst was used in powder form and mixed directly with the biomass feedstock prior to pyrolysis. The reactor was then heated under in-situ conditions. The heating rate in the reactor was approximately 6–10°C/min. Once the target pyrolysis temperature was reached, the temperature was maintained until pyrolysis was complete, as indicated by the cessation of bio-oil production. The total time required to collect no further bio-oil was approximately 2 hours. Each treatment was replicated twice.

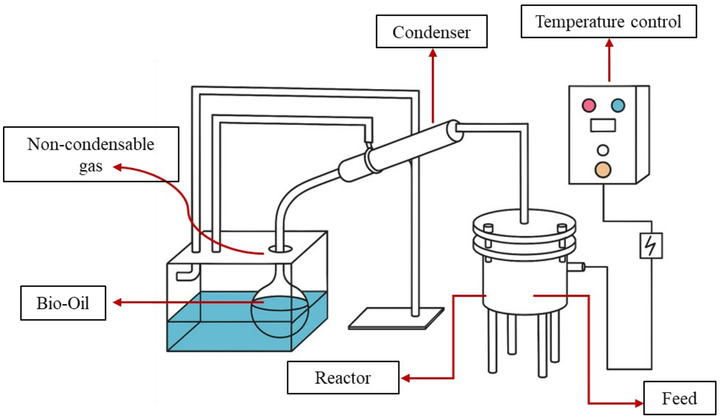

**Figure 1**. Schematic of the pyrolysis reactor

The dependent variables in this study included the bio-oil yield, density, viscosity, and chemical characterization using Fourier Transform Infrared Spectroscopy (FTIR). The research method involved pyrolysis of candlenut shells and coffee husks, each with a total mass of 300 g. The pyrolysis process was carried out in two variations, namely based on the type of feedstock and the use of a catalyst. In the first variation, pyrolysis was performed without catalyst addition, while in the second variation, a $NiCl_2$ catalyst was used at a 3:1 ratio, with 100 g of catalyst introduced into the reactor along with the biomass feedstock. The $NiCl_2$ catalyst was mixed with the biomass at a catalyst-to-biomass mass ratio of 3:1. This ratio was selected based on preliminary experiments and previous studies, which indicated that it provides sufficient catalytic activity to promote vapor-phase reactions and bio-oil formation without inducing excessive secondary cracking or operational constraints.

The pyrolysis temperatures were varied at 250°C, 350°C, and 450°C. During heating, the pyrolysis vapors were passed through a water-cooled condenser at approximately 13°C, with ice blocks added to maintain the temperature. The condensed vapors produced bio-oil, which was collected in a round-bottom flask attached to the end of the condenser. Meanwhile, the non-condensable gases were released from the system after passing through the bio-oil collection flask. The solid residue (char) remained in the reactor as the by-product of the pyrolysis process. All pyrolysis experiments were conducted in duplicate under identical operating conditions. The deviations between repeated runs were minimal, confirming good experimental reproducibility. Therefore, the reported values represent consistent trends and reliable experimental results.

## 2.3. Characterization and Analysis

### 2.3.1. Viscosity

Viscosity can be defined as the ability of a substance to flow through a particular medium. One common method for determining the viscosity of a liquid is using an Ostwald viscometer. The measurement principle involves comparing two types of fluids, specifically distilled water and the liquid sample, with viscosities denoted as $\eta_a$ and $\eta_x$, respectively. Both fluids are prepared with equal volumes and are allowed to flow through capillary tubes of identical dimensions. Since the two fluids possess the same volume but different viscosities, their flow rates will differ, resulting in different flow times required to pass the same volume through the viscometer [11]. The viscosity of the tested liquid can then be calculated using Equation (1).

$$\eta_x = \frac{t_x \times \rho_x \times \eta_a}{t_a \times \rho_a} \tag{1}$$

where, $\eta_a$ is the viscosity of water (cP), $\eta_x$ is the viscosity of the tested liquid (cP), $t_a$ is the flow time of water (s), $t_x$ is the flow time of the tested liquid (s), $\rho_a$ is the density of water (kg/m$^3$), and $\rho_x$ is the density of the tested liquid (kg/m$^3$).

### 2.3.2. Density

The density of bio-oil was analyzed using a 25 mL pycnometer and subsequently calculated based on Equation (2) in accordance with ISO 4787 standards. To minimize measurement errors, all measurements were performed in triplicate for each sample, and the results were reported as averages [12].

$$\rho = \frac{m}{v} \tag{2}$$

where, m is the mass of the sample (g), and v is the volume of the pycnometer (mL).

### 2.3.3. Yield

Yield is the ratio of production inputs to production output and reflects the level of production efficiency. The definition of efficiency in an industry is the ability to produce maximum output with a given amount of input, or to produce a specific amount of output with minimal input. A higher output ratio indicates greater efficiency. In other words, efficiency can be defined as the optimal utilization of input to produce output [2].

$$Mass\ of\ Bio\ oil = Vol.of\ Bio\ oil\ \times Density\ of\ Bio\ oil \tag{3}$$

$$Mass\ of\ Gas = Mass\ of\ Feed - Mass\ of\ Bio\ oil - Mass\ of\ Char \tag{4}$$

$$Bio\ oil\ Yield\ (\%) = \frac{Mass\ of\ Bio\ oil\ (g)}{Mass\ of\ Feed\ (g)} \times 100 \tag{5}$$

$$Char\ Yield\ (\%) = \frac{Mass\ of\ Char\ (g)}{Mass\ of\ Feed\ (g)} \times 100 \tag{6}$$

$$Gas\ Yield\ (\%) = 100 - Bio\ oil\ Yield\ \ - Yield\ Char \tag{7}$$

In this study, the efficiency of the pyrolysis process was evaluated based on the product yields of bio-oil, char, and gas. The mass of bio-oil produced was first determined from the measured volume and density of the bio-oil as expressed in Equation (3). The mass of non-condensable gas was then calculated using an overall mass balance as shown in Equation (4). Furthermore, the percentage yields of bio-oil and char relative to the feed mass were calculated using Equations (5) and (6), respectively. Finally, the gas yield was estimated by difference from the total product distribution, as shown in Equation (7).

### 2.3.4. Mass Balance

The law of conservation of mass, also known as Lavoisier's Law, states that the mass of a closed system remains constant despite various processes occurring within it. For yield determination and mass balance analysis, the catalyst was assumed to be inert and did not participate in the reaction; it only enhanced the reaction rate without undergoing chemical transformation or mass change. Consequently, the catalyst was excluded from the yield and mass balance calculations. The mass of biomass feedstock used in each experiment was 300 g, and the catalyst mass was not considered. When no chemical reactions occur in the system, the mass balance only involves input and output streams. The general mass balance relationship is expressed in Equation (8).

$$Mass\ In - Mass\ Out + Mass\ Generation - Mass\ Consumption = Accumulation \tag{8}$$

It was assumed that no mass generation or mass consumption occurred in the system; therefore, the accumulation term was zero. Under these assumptions, the general mass balance equation in Equation (8) can be simplified to Equation (9). By rearranging Equation (9), the relationship between the inlet and outlet mass flow can be expressed as shown in Equation (10). This indicates that the total mass entering the system must be equal to the total mass leaving the system. Consequently, the steady-state mass balance for the system can be written as in Equation (11), which states that the mass input equals the mass output.

$$Mass\ In - Mass\ Out\ + 0 - 0 = 0 \tag{9}$$

$$Mass\ In - Mass\ Out = 0 \tag{10}$$

$$Mass\ In = Mass\ Out \tag{11}$$

### 2.3.5. FTIR (Fourier Transform Infrared)

The functional groups of the raw materials were analyzed by Fourier transform infrared spectroscopy (FTIR) using a Cary 630 FTIR Spectrometer (Agilent Technologies, Santa Clara, California, USA). Meanwhile, bio-oil samples corresponding to the maximum bio-oil yield achieved under both non-catalytic and catalytic pyrolysis conditions for each feedstock were selected for further characterization. These samples were analyzed using FTIR to identify the functional groups present in the bio-oil. The analysis was performed using an Invenio-R FTIR spectrometer (Bruker), which provides high-resolution spectra suitable for detailed chemical characterization. FTIR spectra of the bio-oil samples were recorded using an FTIR spectrometer at a resolution of 4 $cm^{-1}$ with 32 scans per sample. These parameters were chosen to ensure an adequate signal-to-noise ratio and spectral data reproducibility.

## 3. Results and Discussion

### 3.1. Results of Pyrolysis Experiments on Candlenut Shells and Coffee Shells

In this study, the pyrolysis of candlenut shells and coffee shells produced three types of products: a liquid product known as bio-oil, a solid residue referred to as char, and non-condensable gases. The results of the pyrolysis experiments for both feedstocks are presented in Table 1, which shows the amount of feed (300 g) introduced into the batch reactor at each temperature condition (250, 350, and 450°C). Table 1 summarizes the experimental results of the pyrolysis of Candlenut Husk (CKa) and Coffee Husk (CKo), focusing on the influence of reaction temperature (250°C, 350°C, and 450°C) and the presence of a $NiCl_2$ catalyst on the distribution of bio-oil, char, and gas products.

**Table 1**. Product distribution from the pyrolysis of candlenut shells and coffee shells

| Feedstocks | Conditions | Temperature Conditions (°C) | Replication | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | 1 | | | 2 | | |
| | | | Bio-oil (mL) | Char (g) | Gas (%) | Bio-oil (mL) | Char (g) | Gas (%) |
| Candlenut Shells (CKei) | Non-catalytic $NiCl_2$ | 250 | 96 | 179 | 8 | 88 | 180 | 11 |
| | | 350 | 114 | 152 | 11 | 112 | 150 | 12 |
| | | 450 | 116 | 141 | 14 | 116 | 144 | 13 |
| | Catalytic $NiCl_2$ | 250 | 138 | 104 | 19 | 130 | 109 | 20 |
| | | 350 | 152 | 86 | 20 | 146 | 88 | 22 |
| | | 450 | 164 | 61 | 24 | 160 | 68 | 22 |
| Coffee Shells (CKo) | Non-catalytic $NiCl_2$ | 250 | 110 | 118 | 24 | 122 | 115 | 21 |
| | | 350 | 116 | 101 | 27 | 128 | 100 | 24 |
| | | 450 | 118 | 94 | 29 | 130 | 96 | 24 |
| | Catalytic $NiCl_2$ | 250 | 148 | 115 | 12 | 144 | 121 | 11 |
| | | 350 | 176 | 70 | 18 | 172 | 76 | 17 |
| | | 450 | 164 | 65 | 24 | 168 | 68 | 21 |

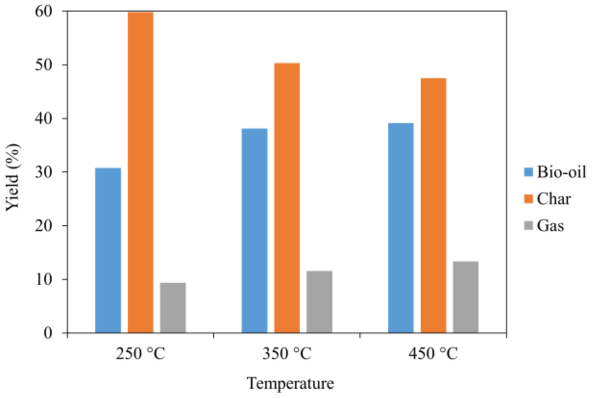

**Figure 2**. Percentage yield of pyrolysis products from candlenut shell

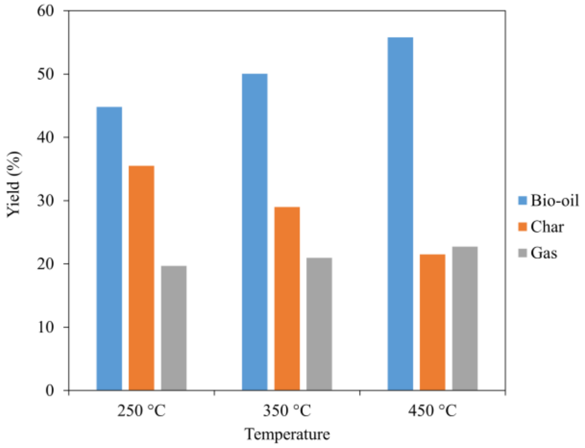

**Figure 3**. Percentage yield of pyrolysis products from candlenut shell using $NiCl_2$ catalyst

All experimental conditions were performed in duplicate to assess reproducibility. The yields obtained from the first and second runs show only slight variations and follow identical distribution trends across all temperatures and catalytic conditions. This consistency indicates good experimental reliability and confirms that the observed differences in product yields are primarily governed by temperature and catalytic effects rather than experimental uncertainty. Consequently, the data presented in Table 1 represent both the experimental outcomes and their replications and serve as a reliable basis for the subsequent analysis and discussion of product distribution behavior. Figures 2 to 5 present the average values from the two replications shown in Table 1 and are used to clarify the trends in product distribution with respect to temperature and catalyst usage.

A clear trend was observed in which increasing the process temperature consistently decreased the yield of solid char while enhancing gas production, primarily due to more extensive thermal decomposition and secondary cracking reactions. The addition of $NiCl_2$ catalyst proved highly effective, markedly improving biomass conversion by significantly reducing char formation and increasing the yields of both bio-oil and gas compared to the non-catalytic process. The close consistency between the two replicates for each experimental condition further confirms the reliability of these findings. The data presented in the table were averaged, and the calculated yields of gas, bio-oil, and char are illustrated in the following graph.

Based on Figures 2 and 3, the pyrolysis of candlenut shell and the catalytic pyrolysis of candlenut shell with $NiCl_2$ show a similar trend, where increasing the pyrolysis temperature results in higher yields of bio-oil and gas, while the yield of solid char decreases. This occurs because at higher temperatures, more of the raw material undergoes thermal degradation, leading to greater conversion into volatile compounds and a lower solid residue. This phenomenon is influenced by the lignocellulosic content present in the candlenut shell. Candlenut shell contains 49.22% holocellulose and 54.46% lignin [13]. However, another reference reports hemicellulose, cellulose, and lignin contents of 33%, 17%, and 34%, respectively [14]. The high lignin content in candlenut shell affects its degradation temperature, as lignin decomposes at around 410°C, while cellulose and hemicellulose degrade at approximately 350°C and 300°C [15]. Therefore, pyrolysis at 450°C in this study produced the highest bio-oil yield, as most lignocellulosic components were decomposed at this temperature. The application of a catalyst during the pyrolysis of candlenut shells significantly influences product yield distribution, particularly enhancing the formation of bio-oil and gaseous products.

As illustrated in Figures 1 and 2, the incorporation of $NiCl_2$ catalyst significantly increased the bio-oil yield, indicating improved catalytic cracking and secondary decomposition of volatile intermediates. In contrast, the yield of solid char declined markedly following the introduction of $NiCl_2$, suggesting a higher degree of feedstock conversion during catalytic pyrolysis. The role of $NiCl_2$ as a catalyst is selective in promoting bio-oil formation and accelerating the degradation of candlenut shells. The presence of this catalyst enhances the pyrolysis reaction rate, leading to a more efficient process and a higher bio-oil yield. As the degradation process intensifies, the organic components of the feedstock are converted into volatile compounds, leaving behind carbonaceous residues. Consequently, at higher pyrolysis temperatures, more extensive degradation of organic matter occurs, leading to lower yields of solid products. The use of $NiCl_2$ catalyst can enhance the yield of bio-oil in the pyrolysis of oil palm fiber compared to the process without a catalyst. $NiCl_2$ lowers the activation energy required to break the chemical bonds of organic compounds in biomass, thereby accelerating the pyrolysis reaction rate and increasing bio-oil production.

The pyrolysis of candlenut and coffee shells in the absence of a catalyst showed that increasing the pyrolysis temperature significantly increased the yields of bio-oil and gaseous products, while the char yield decreased. The optimum bio-oil yields obtained from candlenut and coffee shells were 39.14% and 41.80%, respectively, at 450°C. This behavior can be attributed to the complete thermal decomposition of the major biomass constituents (cellulose, hemicellulose, and lignin) at elevated temperatures. Specifically, hemicellulose decomposes at 220–315°C, cellulose at 315–400°C, and lignin over a broader range of 400–550°C [16]. The enhanced secondary cracking observed for catalytic coffee shell pyrolysis above 350°C is likely related to its higher ash and alkali metal content (e.g., K, Ca, Mg), which can act as intrinsic catalysts and interact synergistically with $NiCl_2$, promoting vapor cracking and gas formation at elevated temperatures. In contrast, candlenut shells generally have lower ash content and higher lignin fractions; lignin-derived volatiles are more thermally stable and less prone to secondary cracking even at 450°C. Thus, the different secondary cracking behaviors reflect the combined effects of biomass composition and the interaction between intrinsic minerals and the $NiCl_2$ catalyst.

Pyrolysis of coffee shells without a catalyst showed that increasing temperature led to higher yields of bio-oil and gas, while the solid residue decreased. This result is consistent with previous studies on coffee shell pyrolysis, where an increase in temperature corresponded to a higher bio-oil yield [17]. This behavior is attributed to the substantial cellulose content in coffee shells, which undergoes thermal degradation and contributes significantly to bio-oil formation [18]. The chemical composition analysis of coffee shells revealed that they contain approximately 37.3% cellulose, 17.02% hemicellulose, and 26.44% lignin. In addition, the remaining constituents comprise around 5% ash and 19.28% extractive substances [19].

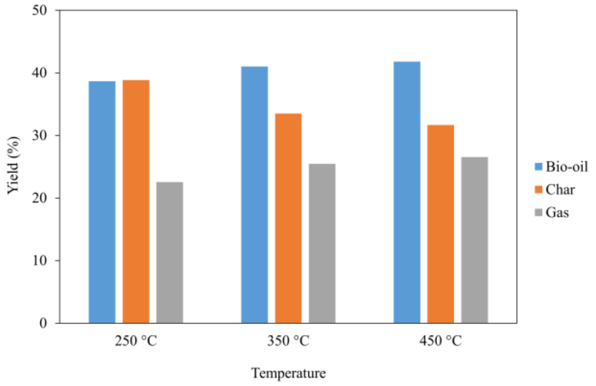

**Figure 4**. Percentage yield of coffee husk pyrolysis products

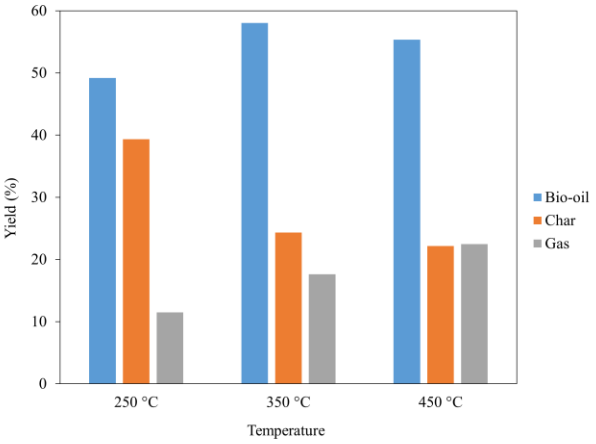

**Figure 5**. Percentage yield of coffee husk pyrolysis products with $NiCl_2$ catalyst

Upon introduction of the catalyst, the product distribution showed a distinct trend. The optimum bio-oil yields of candlenut and coffee shells with catalyst addition were 55.78% at 450°C and 58.05% at 350°C, respectively, after which the yields decreased to 55.36% at 450°C. The higher holocellulose content in coffee shells compared to lignin explains why catalytic pyrolysis produced more bio-oil at 350°C than at 450°C. Cellulose and hemicellulose mainly decompose below 400°C, and the catalyst accelerates these degradation reactions, making 350°C the optimal temperature. In contrast, for candlenut shells, bio-oil yield continued to increase with increasing pyrolysis temperature under catalytic conditions. This phenomenon is attributed to the higher lignin content in candlenut shells, resulting in a maximum bio-oil yield at 450°C, when lignin undergoes significant thermal decomposition.

The optimum bio-oil yield obtained with the addition of a catalyst was higher than that achieved without a catalyst for both candlenut and coffee shells. This enhancement can be attributed to the catalytic effect. The difference in the optimum temperature for maximum bio-oil yield between coffee shells and candlenut shells is primarily related to their distinct biomass compositions and mineral contents. Coffee shells are richer in holocellulose and intrinsic alkali minerals, which decompose and volatilize at lower temperatures. In the presence of $NiCl_2$, these characteristics promote rapid primary cracking and enhanced vapor-phase reactions at around 350°C, resulting in a maximum bio-oil yield.

At higher temperatures, secondary cracking becomes dominant, increasing gas formation and reducing bio-oil yield. In contrast, candlenut shells contain more lignin, which requires higher temperatures for effective depolymerization. The catalytic activity of $NiCl_2$ becomes more effective at elevated temperatures (~450°C) for lignin-rich feedstocks, facilitating the breakdown of complex aromatic structures into condensable bio-oil compounds without excessive secondary cracking. Consequently, the maximum bio-oil yield for candlenut shells is achieved at 450°C, whereas coffee shells reach their optimum at a lower temperature.

A catalyst is a chemical substance that accelerates a reaction toward equilibrium without altering the chemical structure of the products at the end of the reaction. Although it does not change the equilibrium position, the catalyst functions by lowering the activation energy, thereby reducing the minimum energy required for effective molecular collisions and enabling the reaction to proceed more rapidly [20].

### 3.2. Mass Balance of Candlenut Shell and Coffee Shell Pyrolysis With and Without Catalyst

The mass balance in this study represents the calculation of the input and output masses from the pyrolysis process. The input mass consists of the initial raw materials, namely candlenut shells and coffee shells, with or without the addition of a catalyst, while the output mass refers to the products obtained. Products from the pyrolysis of candlenut and coffee shells, with and without a catalyst, include bio-oil, char, and gas.

**Table 1**. Mass balance of candlenut shell without catalyst

| Compositions | 250°C | | 350°C | | 450°C | |
|---|---|---|---|---|---|---|
| | Input (g) | Output (g) | Input (g) | Output (g) | Input (g) | Output (g) |
| Candlenut shell | 300 | - | 300 | - | 300 | - |
| Bio-oil | - | 92 | - | 113 | - | 116 |
| Char | - | 179.5 | - | 151 | - | 142.5 |
| Gas | - | 28.5 | - | 36 | - | 41.5 |
| Total | 300 | 300 | 300 | 300 | 300 | 300 |

**Table 2**. Mass balance of coffee shell without a catalyst

| Compositions | 250°C | | 350°C | | 450°C | |
|---|---|---|---|---|---|---|
| | Input (g) | Output (g) | Input (g) | Output (g) | Input (g) | Output (g) |
| Coffee shell | 300 | - | 300 | - | 300 | - |
| Bio-oil | - | 116 | - | 122 | - | 124 |
| Char | - | 116.5 | - | 100.5 | - | 95 |
| Gas | - | 67.5 | - | 77.5 | - | 81 |
| Total | 300 | 300 | 300 | 300 | 300 | 300 |

**Table 3**. Mass balance of candlenut shell with catalyst

| Compositions | 250°C | | 350°C | | 450°C | |
|---|---|---|---|---|---|---|
| | Input (g) | Output (g) | Input (g) | Output (g) | Input (g) | Output (g) |
| Candlenut shell | 300 | - | 300 | - | 300 | - |
| Bio-oil | - | 134 | - | 149 | - | 162 |
| Char | - | 106.5 | - | 87 | - | 64.5 |
| Gas | - | 59.5 | - | 64 | - | 73.5 |
| Total | 300 | 300 | 300 | 300 | 300 | 300 |

**Table 4**. Mass balance of coffee shell with catalyst

| Compositions | 250°C | | 350°C | | 450°C | |
|---|---|---|---|---|---|---|
| | Input (g) | Output (g) | Input (g) | Output (g) | Input (g) | Output (g) |
| Coffee shell | 300 | - | 300 | - | 300 | - |
| Bio-oil | - | 146 | - | 174 | - | 166 |
| Char | - | 118 | - | 73 | - | 66.5 |
| Gas | - | 36 | - | 53 | - | 67.5 |
| Total | 300 | 300 | 300 | 300 | 300 | 300 |

The mass balance in this study represents the equilibrium between the input and output masses within the reactor system. The feed introduced into the reactor, both with and without the addition of a catalyst, was 300 g, and the total mass of the resulting products was also 300 g. The input materials consisted of candlenut shells and coffee shells, with or without the presence of a catalyst, while the output components were bio-oil, char, and gas. The catalyst did not undergo any significant change during the process; therefore, the amount of catalyst entering the system was equal to that leaving it.

Based on the mass balance results, the conversion efficiency of biomass to liquid products (bio-oil) increased with increasing pyrolysis temperature and with the use of a catalyst. In the absence of a catalyst, the solid (char) fraction remained relatively high, indicating incomplete decomposition of the biomass components. However, the introduction of the $NiCl_2$ catalyst enhanced the conversion of organic matter into bio-oil and gaseous products by lowering the activation energy of the pyrolysis reactions. This finding demonstrates that the catalyst effectively promotes the decomposition of cellulose, hemicellulose, and lignin into volatile compounds, which subsequently condense to form bio-oil. Overall, the use of a catalyst improved both the process efficiency and the bio-oil yield compared to non-catalytic pyrolysis.

### 3.3. Density and Viscosity of Bio-Oil

Density is a measure of the compactness of a liquid, defined as the ratio between the mass of the liquid and its volume. The density test was conducted to determine the mass of a material per unit volume [21]. Based on the experimental results, the density values of the bio-oil produced from the pyrolysis process in this study are presented in Table 5.

As the pyrolysis temperature increases for both candlenut and coffee shells, with or without a catalyst, the resulting bio-oil density decreases. This phenomenon occurs because higher temperatures promote molecular motion and the breakdown of long carbon chains, leading to simpler molecular structures with lower molecular weights and, consequently, lower densities. Density influences the combustion characteristics of fuel; a lower density generally improves fuel ignition due to its higher calorific value [22]. The density of bio-oil derived from candlenut shells ranged from 1.0025 to 1.0441 g/mL, while that from coffee shells ranged from 0.9983 to 1.0157 g/mL. The highest density was obtained from candlenut shell bio-oil produced with an $NiCl_2$ catalyst at 250°C, measuring 1.0441 g/mL, whereas the lowest density was observed for coffee shell bio-oil without a catalyst at 450°C, measuring 0.9983 g/mL, which is close to the density of water. This indicates a relatively high water content in the sample.

The bio-oil yields obtained in this study, reaching up to 58.05% under catalytic conditions, are consistent with values reported in the literature for catalytic pyrolysis of lignocellulosic biomass. Although the maximum density value recorded (1.0441 g/mL) is relatively high compared to conventional petroleum fuels, it remains within the typical range reported for bio-oil. This elevated density is mainly attributed to the presence of polar oxygenated compounds and residual water formed during dehydration reactions, which also influence viscosity behavior.

The higher bio-oil density obtained from catalytic candlenut shell pyrolysis at 250°C compared to the non-catalytic condition can be attributed to the $NiCl_2$ catalyst's promotion of early-stage reactions at relatively low temperatures. At 250°C, thermal cracking is still limited; however, the catalyst may enhance depolymerization and condensation of lignin-derived intermediates, forming heavier oxygenated compounds and oligomeric species that increase bio-oil density. As the temperature increases, secondary cracking becomes more dominant, breaking down these heavier compounds into lighter fractions, which explains the decrease in density observed at higher temperatures. Thus, the catalyst influences the chemical composition of bio-oil differently at low temperatures, favoring the formation of heavier compounds before extensive cracking. Additionally, the molecular weight of bio-oil components significantly affects its density; a higher molecular weight results in a higher density [23].

Viscosity is a measure that indicates the resistance of a liquid to flow, or in other words, the degree of fluid thickness. The viscosity test is conducted to determine the fluidity or thickness value of a liquid [21]. Based on the experimental results, the viscosity of the bio-oil produced by pyrolysis is presented in Table 5. Viscosity refers to the internal resistance of a fluid to flow or the measure of its thickness [21]. The viscosity of the produced bio-oil is presented in Table 6. Similar to density, viscosity decreases with increasing pyrolysis temperature for both candlenut and coffee shells, with or without a catalyst. This occurs because higher temperatures break long carbon chains into shorter and simpler molecules, resulting in a less viscous liquid [2]. The viscosity of bio-oil obtained from candlenut shells ranged from 0.824 to 0.978 cP, while that from coffee shells ranged from 0.834 to 0.980 cP.

The use of $NiCl_2$ catalyst in the pyrolysis process further reduced the viscosity of bio-oil compared to non-catalytic pyrolysis. This effect is attributed to the catalyst's ability to promote deoxygenation reactions and break down long carbon chains into lighter hydrocarbons. As a result, the bio-oil consists of simpler molecular structures and fewer oxygenated compounds, such as carboxylic acids and phenols, which are known to increase viscosity. Therefore, the application of $NiCl_2$ catalyst not only enhances bio-oil yield but also improves its physical properties, making it more comparable to conventional liquid fuels.

However, it is essential to maintain an optimal viscosity range, as excessively low viscosity may lead to combustion instability and injection leakage, while too high viscosity may cause difficulties in fuel pumping and atomization [24]. From an economic perspective, the use of $NiCl_2$ offers practical advantages, including low cost, commercial availability, and ease of handling, making it an attractive catalyst for biomass pyrolysis at larger scales. When compared with studies on other biomass feedstocks, the observed trends in yield enhancement and product quality are consistent, while also highlighting the importance of biomass composition and catalyst–feedstock interactions in determining optimal operating conditions.

### 3.4. Result of FTIR Analysis

Characterization using FTIR spectroscopy was conducted to identify the functional bonds formed in the coffee shell and candlenut shell samples. The principle of FTIR analysis is to identify functional groups and analyze the composition and structure of the tested samples. The FTIR characterization was performed over the wavenumber range of 400–4000 $cm^{-1}$. In this study, the samples analyzed were those with the highest yields for each type of raw material and catalyst. The FTIR characterization results of the coffee shell, candlenut shell, and their respective bio-oil products, both with and without the $NiCl_2$ catalyst.

**Table 5**. Density and viscosity of bio-oil

| Feedstocks | Conditions | Temperature variation (°C) | Density (g/mL) | Viscosity (cP) |
|---|---|---|---|---|
| Candlenut Shells (CKei)<br>(CKe) | Non-catalytic $NiCl_2$ | 250 | 1.0123 | 0.9712 |
| | | 350 | 1.0114 | 0.9569 |
| | | 450 | 1.0038 | 0.9230 |
| | Catalytic $NiCl_2$ | 250 | 1.0331 | 0.9109 |
| | | 350 | 1.0073 | 0.8781 |
| | | 450 | 1.0032 | 0.8478 |
| Coffee Shells (CKo)<br>(CKo) | Non-catalytic $NiCl_2$ | 250 | 1.0114 | 0.9178 |
| | | 350 | 1.0087 | 0.9088 |
| | | 450 | 0.9994 | 0.8807 |
| | Catalytic $NiCl_2$ | 250 | 1.0103 | 0.9142 |
| | | 350 | 1.0008 | 0.9025 |
| | | 450 | 1.0004 | 0.8858 |

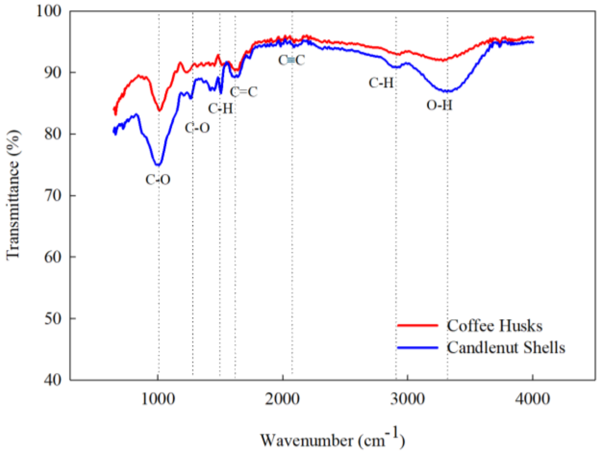

**Figure 6**. FTIR spectra of the raw materials: candlenut husk and coffee shell

Figure 6 presents the FTIR analysis results of the raw materials, candlenut shell, and coffee shell. Overall, both raw materials exhibit similar peak patterns. However, the FTIR spectrum of the candlenut shell shows stronger peak intensities compared to that of the coffee shell. The FTIR spectrum in the range of 900–1300 cm$^{-1}$ indicates the presence of C–O functional groups, which correspond to the main glycosidic structure of cellulose. The band observed at 1400–1500 cm$^{-1}$ represents C–H functional groups, showing methylene vibrations that are part of the cellulose structure [25]. The absorption band at 1580–1600 cm$^{-1}$ corresponds to C=H groups, indicating aromatic structures derived from lignin. The spectrum in the range of 2700–2850 cm$^{-1}$ represents C–H stretching vibrations, while the broad band at 3200–3600 cm$^{-1}$ corresponds to O–H groups, indicating alcoholic structures typical of cellulose [26]. These functional groups are commonly found in all types of biomass.

Figure 7 presents the results of candlenut shell pyrolysis, both with and without a catalyst, at a temperature of 450°C, which is the temperature that yielded the highest bio-oil yield compared to other pyrolysis temperatures. Based on the data (Figure 7), pyrolysis of candlenut shells with a catalyst shows higher intensity than without a catalyst. The use of a catalyst in this pyrolysis process can improve the quality of the bio-oil [27]. Variations in the FTIR spectra of the raw materials and the resulting bio-oil from catalytic and non-catalytic pyrolysis are shown in Figures 8 and 9. Figure 8 presents a comparative analysis of the FTIR spectra within the wavenumber range of 400–2000 cm$^{-1}$, whereas Figure 9 provides a comparison of the spectra in the wavenumber range of 2000–4500 cm$^{-1}$ for both catalytic and non-catalytic pyrolysis-derived bio-oil samples.

The FTIR spectrum of the candlenut shell (Figure 8) displays the typical signatures of lignocellulosic biomass, with prominent carbonyl stretching at 1710–1740 cm$^{-1}$, C–O vibrations within 1020–1220 cm$^{-1}$, and aromatic C=C absorption near 1500–1600 cm$^{-1}$. The presence of aliphatic C–H deformation bands at 1360–1450 cm$^{-1}$ further reflects contributions from cellulose, hemicellulose, and lignin.

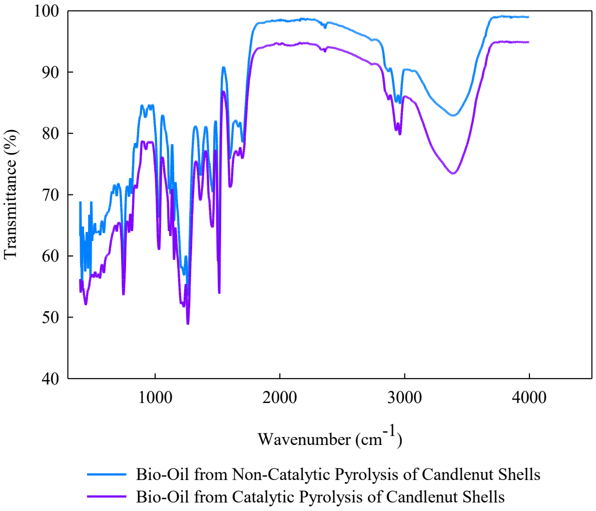

**Figure 7**. FTIR spectra of bio-oil from candlenut shell pyrolysis without a catalyst and with a catalyst

As shown in Figure 9, notable spectral transformations occur in the bio-oil samples produced under catalytic and non-catalytic pyrolysis conditions. A reduction in the C=O band intensity denotes carbonyl degradation during thermal cracking, while alterations in the C–O region indicate the generation of oxygenated species, including phenolic and alcohol derivatives. The broad O–H band at 3200–3600 cm$^{-1}$ becomes more pronounced, consistent with an increased presence of hydroxyl-containing compounds. The catalytic bio-oil exhibits sharper, more distinct absorption features than the non-catalytic product, indicating enhanced structural deconstruction facilitated by the catalyst. The attenuated aromatic C=C band further suggests more extensive ring-opening reactions and a shift toward lighter aliphatic constituents. Collectively, these spectral differences substantiate the role of the catalyst in promoting deeper depolymerization and modifying the molecular profile of the resulting bio-oil.

Figure 9 presents the FTIR spectra of bio-oil obtained from catalytic and non-catalytic pyrolysis of candlenut shells in the higher wavenumber region. Both spectra feature a distinct band at 2850–3000 cm$^{-1}$, assigned to aliphatic C–H stretching vibrations, indicating the presence of hydrocarbon chains formed during thermal decomposition. The non-catalytic bio-oil exhibits a stronger C–H band, suggesting a higher abundance of aliphatic compounds in the absence of the catalyst. A broad absorption band centered at 3200–3600 cm$^{-1}$ corresponds to O–H stretching, which is characteristic of phenolic and alcohol-type oxygenates. The catalytic bio-oil shows a broader and more intense O–H band, indicating increased hydroxyl-containing compounds from catalyst-driven fragmentation and partial deoxygenation. The observed spectral variations between the two samples demonstrate that catalytic pyrolysis substantially alters the functional group distribution in the resulting bio-oil. In particular, the catalyst facilitates structural transformation pathways that enhance the production of oxygenated species while simultaneously reducing the relative contribution of aliphatic C–H groups.

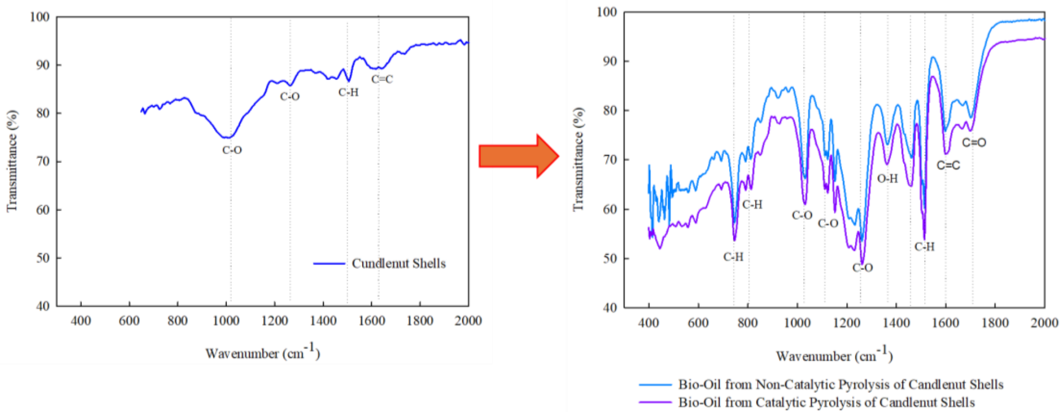

**Figure 8**. FTIR spectra comparison of raw materials and bio-oil from catalytic and non-catalytic candlenut shell pyrolysis (400–2000 cm$^{-1}$)

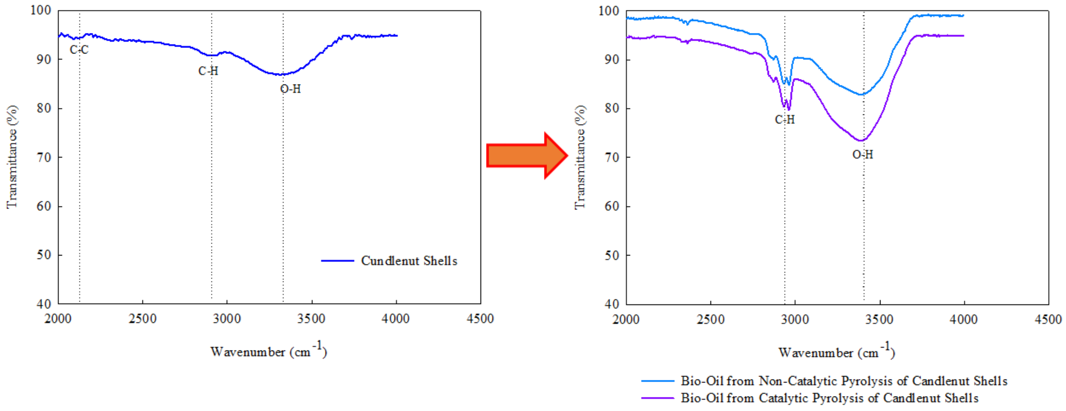

**Figure 9**. FTIR spectra comparison of raw materials and bio-oil from catalytic and non-catalytic candlenut shell pyrolysis (2000–4500 cm$^{-1}$)

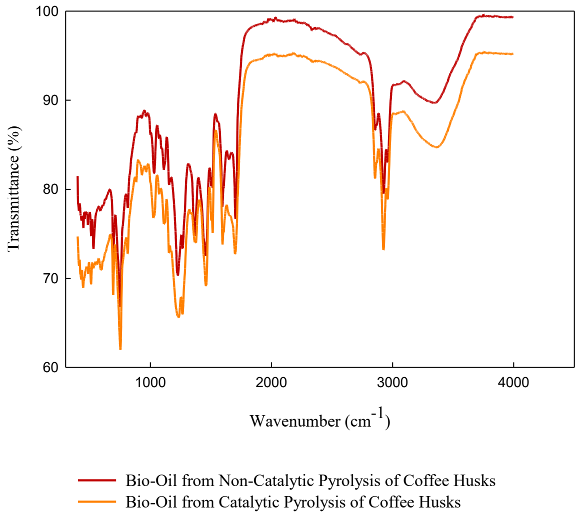

**Figure 10**. FTIR spectra of bio-oil from coffee shell pyrolysis without and with a catalyst

Figure 10 shows the FTIR spectra of bio-oil produced from catalytic and non-catalytic pyrolysis of coffee husks exhibit several characteristic absorption features indicative of the chemical transformations occurring during thermal conversion. Both samples show multiple bands in the 1000–1700 cm$^{-1}$ region, corresponding to C–O, C=C, and C–H vibrations commonly associated with phenolic, carbonyl, and aromatic structures—reflecting the decomposition of lignocellulosic components present in the raw biomass. A pronounced absorption centered around ~1700 cm$^{-1}$ is attributed to C=O stretching, indicating the presence of carbonyl-containing compounds such as ketones, aldehydes, and carboxylic acids. This band appears slightly more intense in the catalytic bio-oil, suggesting that catalytic reactions promote additional fragmentation pathways leading to oxygenated species.

The broad feature near 3000–3500 cm$^{-1}$ corresponds to O–H stretching vibrations, typically associated with alcohols and phenolic groups. The catalytic bio-oil spectrum shows a broader and deeper O–H band, indicating a higher concentration of hydroxyl-containing compounds, likely due to catalyst-facilitated breakdown of lignin structures. Furthermore, spectral differences between the two samples, particularly in the 1000–1500 cm$^{-1}$ region, suggest that catalytic pyrolysis enhances cracking reactions, resulting in a more complex mixture of oxygenates and aromatic derivatives. The reduced transmittance in this region for the catalytic bio-oil supports the formation of compounds with stronger infrared absorption, consistent with the catalytic modification of pyrolysis pathways.

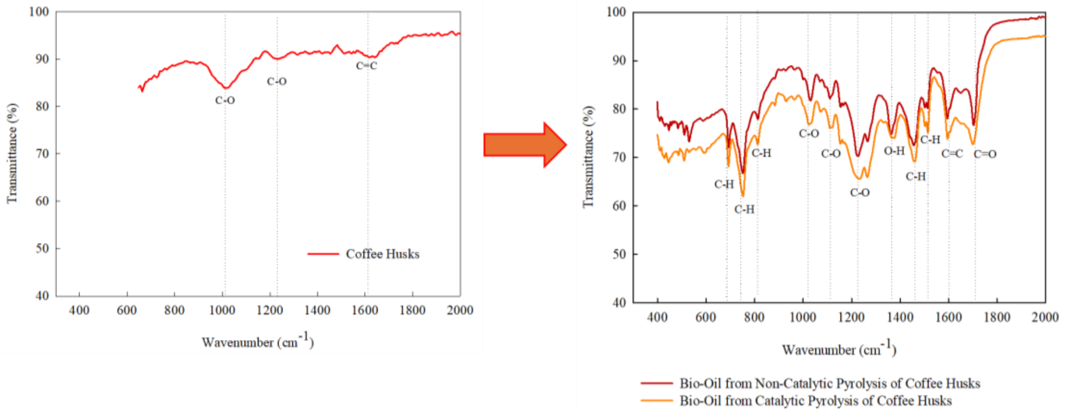

**Figure 11**. Comparative FTIR spectra of the raw coffee shell feedstock and the resulting bio-oil obtained from catalytic and non-catalytic pyrolysis within the 400–2000 cm$^{-1}$ region

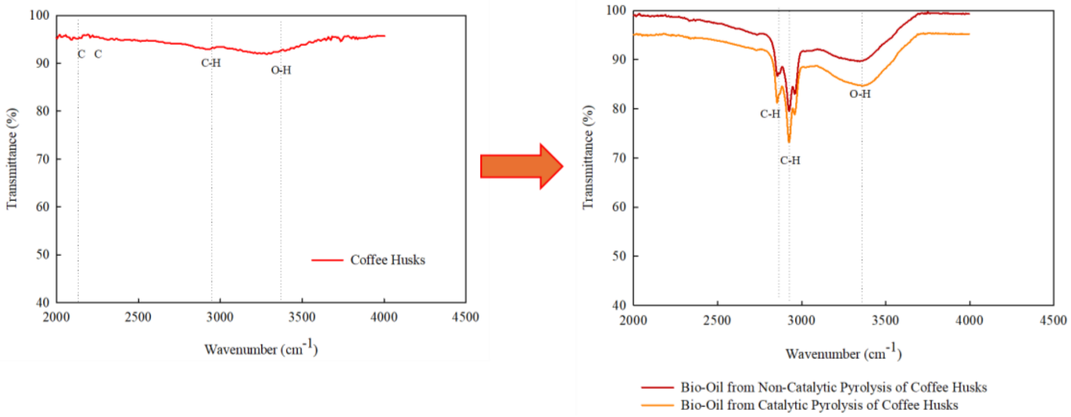

**Figure 12**. Comparative FTIR spectra of the raw coffee shell feedstock and the resulting bio-oil obtained from catalytic and non-catalytic pyrolysis within the 2000-4500 cm$^{-1}$ region

Based on Figure 11, the spectrum of raw coffee husks exhibits key lignocellulosic signatures, including C=O stretching near ~1730 cm$^{-1}$, C–O vibrations in the 1000–1250 cm$^{-1}$ range, and aromatic C=C absorption around ~1600 cm$^{-1}$, confirming the presence of cellulose, hemicellulose, and lignin. In contrast, the spectra of the bio-oils show pronounced modifications resulting from thermal cracking. The intensification and broadening of the C–O and C–H bands, particularly in the 700–1500 cm$^{-1}$ region, indicate the formation of a more complex mixture of phenolic, carbonyl, and aliphatic compounds. Notably, catalytic bio-oil shows sharper, more distinct peaks, suggesting enhanced cracking and fragmentation facilitated by catalytic reactions. The catalytic bio-oil also shows reduced aromatic C=C intensity compared to its non-catalytic counterpart, indicating more extensive depolymerization and ring-opening reactions. These spectral variations confirm that catalytic pyrolysis alters the distribution of oxygenated compounds and promotes deeper structural degradation of the biomass.

Furthermore, in the higher wavenumber region (Figure 12), the raw coffee husks display characteristic aliphatic C–H stretching bands around 2850–3000 cm$^{-1}$, along with a broad O–H stretching band near 3200–3600 cm$^{-1}$, which is typical of moisture, alcohols, and phenolic hydroxyl groups in lignocellulosic materials. Both bio-oil samples exhibit a significant increase in intensity of the C–H stretching bands, indicating the generation of hydrocarbons during pyrolysis. The catalytic bio-oil exhibits a deeper, more distinct C–H band, suggesting improved cracking performance. A broad and enhanced O–H stretching band is observed in both bio-oils, with a notably stronger presence in the catalytic bio-oil. This enhancement indicates a higher concentration of hydroxyl-containing oxygenates, likely from catalytic cleavage of lignin structures, resulting in increased formation of phenolic and alcohol-derived compounds.

The FTIR spectra provide further insight into the chemical evolution of bio-oil under catalytic conditions. While FTIR analysis is inherently qualitative, comparative evaluation of peak intensities reveals notable differences between catalytic and non-catalytic samples, particularly in oxygen-containing functional groups such as O–H, C–O, and C=O. The sharper and more intense absorption bands observed in catalytic bio-oil indicate that $NiCl_2$ influences reaction pathways by promoting selective dehydration and controlled cracking rather than extensive deoxygenation. The sharper, more distinct absorption features observed in the FTIR spectra of the catalytic bio-oil indicate a more selective formation of specific functional groups in the presence of $NiCl_2$. The increased intensity of O–H stretching bands suggests an enrichment of phenolic –OH groups, which are commonly associated with lignin-derived compounds. This implies that $NiCl_2$ does not primarily promote complete deoxygenation at the studied conditions but rather influences the reaction pathways toward selective bond cleavage and transformation.

As a Lewis acid catalyst, $NiCl_2$ can coordinate with oxygen-containing functional groups, facilitating dehydration and depolymerization reactions of biomass constituents. At moderate temperatures, this coordination may promote dehydration reactions that form unsaturated structures (C=C and C≡C), while simultaneously stabilizing phenolic intermediates before extensive cracking occurs. Consequently, the catalytic process favors the formation of oxygenated aromatic compounds rather than fully deoxygenated hydrocarbons. Therefore, the FTIR observations suggest that $NiCl_2$ enhances reaction selectivity by directing the pyrolysis pathways toward dehydration and controlled cracking mechanisms, leading to a bio-oil enriched in phenolic and unsaturated compounds.

## 4. Conclusion

This study examined the effects of feedstock type, pyrolysis temperature, and $NiCl_2$ catalysis on bio-oil production from candlenut shells and coffee shells. Temperature strongly influenced product distribution, while the catalytic effect depended on the biomass type. Bio-oil yield from candlenut shells increased with temperature, whereas catalytic pyrolysis of coffee shells showed a maximum yield at 350°C, followed by a decrease at 450°C due to secondary cracking. Increasing temperature reduced bio-oil density and viscosity for both feedstocks, indicating enhanced thermal cracking and formation of lighter compounds. FTIR analysis revealed dominant oxygen-containing functional groups, confirming that $NiCl_2$ promotes dehydration and controlled cracking rather than complete deoxygenation. The resulting bio-oil is suitable for use as a low- to medium-grade heating fuel and has potential for further upgrading. Despite limitations in catalyst use and bio-oil upgrading, catalytic pyrolysis of coffee shells at 350°C yielded the best overall performance, underscoring the importance of aligning catalyst characteristics with biomass composition for effective lignocellulosic waste valorization.

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