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Bio-oil production from cotton fabric and plastic waste: influence of pyrolysis time in catalytic co-pyrolysis

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ABSTRACT

This study investigates the influence of pyrolysis time on product yield and bio-oil quality in the catalytic co-pyrolysis (CCP) of cotton fabric waste (CFW) and polypropylene plastic waste (PPW). The experiments were conducted in a fixed-bed microreactor using a chromium-extracted alumina (15CE) catalyst, with reaction times varied between 15 and 75 min under constant conditions (500 °C, 1:1 CFW/PPW ratio, and 1:1 F/C ratio). The results indicate that pyrolysis time significantly affects bio-oil and gas production. Extending the reaction time to 60 min maximized bio-oil yield, reaching 76.4 %. The minimal variation in char yield across different pyrolysis times suggests that the thermal degradation of the feedstock was completed within the first 15 min of CCP. GC-MS analysis revealed that bio-oil primarily consists of hydrocarbon and alcohol, while the concentrations of acids and sugar-derived compounds decreased, indicating their conversion into valuable fuel and chemical products. At a reaction time of 75 min, the formation of aromatic hydrocarbons increased, accompanied by a reduction in oxygenated compounds. Based on both bio-oil yield and quality, a pyrolysis time of 60 min was identified as the best condition for the catalytic co-pyrolysis of CFW-PPW using the 15CE catalyst, achieving the highest yield while maintaining an acceptable bio-oil quality.

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1. INTRODUCTION

The growing buildup of waste materials, especially plastic and textile waste, has become a major environmental and societal concern. Plastic waste, made up of polypropylene (PP) and other polymers, is particularly susceptible to degradation, but textile waste, such as cotton fabric, frequently ends up in landfills or burns, adding to pollution and resource waste. Pyrolysis, a thermochemical process that decomposes organic molecules in an oxygen-free atmosphere, appears promising to address these issues. Pyrolysis facilitates the conversion of polyolefins and biomass materials into valuable feedstocks, offering several potential benefits, including waste volume reduction, chemical recovery, and the substitution of fossil fuels [1]. Pyrolysis, which breaks down complex waste materials into simpler molecules at elevated temperatures, turns trash into valuable resources, including liquid oil, char, and gas. Catalytic co-pyrolysis (CCP) is the simultaneous pyrolysis of mixed feedstocks, such as plastics and biomass, with the application of catalysts.

Catalytic co-pyrolysis involves two main reaction pathways: primary and secondary reactions, which correspond to thermal and catalytic processes, respectively. The co-pyrolysis of biomass and plastics has been extensively explored, with most studies demonstrating the favourable constructive interaction between both feedstocks in terms of hydrocarbon and aromatic selectivity [2–4]. Primary volatiles produced during the process undergo further reactions, resulting in the creation of more selective compounds via catalytic cracking [5]. In the catalytic co-pyrolysis (CPP) process, the feedstock is heated to an elevated temperature and held there for a specified duration of time. This condition controls the extent of pyrolysis reactions [6], which substantially impacts both primary and secondary reactions by changing mass and heat transfer dynamics inside the reactor [7]. Subsequently, determining the composition of the pyrolysis products.

Pyrolysis time plays a significant role during the pyrolysis process. Too short pyrolysis time may lead to incomplete feedstock conversion and reduce bio-oil yield. On the contrary, longer pyrolysis time causes excessive cracking and increases the gas yield [8]. Optimization of pyrolysis times, considering a broad range of variables, may help obtain better quality liquid oils [7]. Zhang et al. [9] found that the olefin, aromatics and coke reduced significantly by shortening the retention time of feedstock. Meanwhile, Awang and co-workers [10] concluded that pyrolysis time significantly influences higher temperatures rather than lower temperatures. It is also highlighted from the study by Miandan et al. [11] that the bio-oil contains different aromatic compositions at different pyrolysis times when polystyrene was pyrolyzed at 450 °C and the time between 60 and 120 min. Similar findings were obtained by Nabeel et al. [12] in the CCP process of biomass and plastic, where increased pyrolysis time demonstrates a positive impact on bio-oil yield. However, beyond this duration, it showed minimal impact.

While pyrolysis reaction time plays a crucial role in determining bio-oil yield and quality, research on this aspect remains scarce, particularly in catalytic co-pyrolysis systems. Existing studies primarily focus on feedstock composition, catalyst selection, and temperature effects, leaving a significant knowledge gap regarding the precise influence of pyrolysis time on product distribution and composition. Specifically, limited research was conducted on the catalytic co-pyrolysis of cotton fabric waste (CFW) and polypropylene waste (PPW) using chromium-extracted alumina catalysts. Given the potential of this catalyst to enhance bio-oil yield and selectivity, further investigations are necessary to understand optimal pyrolysis times, degradation pathways, and catalyst interactions. Addressing this gap will contribute to the development of more efficient and sustainable pyrolysis processes. In our previous study, the CCP of CFW and PPW using chromium-extracted Aluminum (CE) as a catalyst with varying metal ratios demonstrated significant potential for bio-oil production [13]. Thus, the present study investigated the influence of pyrolysis time on product distribution and quality. Additionally, the functional group characteristics of resulting products were analysed to understand the chemical composition and catalytic effect further.

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2. MATERIALS AND METHOD

2.1 Materials

The chemicals used in the catalyst preparation were chromium (III) nitrate nonahydrate with 98 wt.% purity and sulfuric acid with 97 wt.% purity, which were purchased from Qrec Malaysia. Meanwhile, ammonium solution (purity 30 wt.%) was purchased from R&M Malaysia. The sludge and cotton fabric waste (CFW) were collected from local industries, while polypropylene plastic waste (PPW) was collected from residential areas.

2.2 Method

The catalyst of chromium-extracted aluminum (CE) was synthesized by extracting the Aluminum from sludge using acid-leaching techniques. A 250 mL of 2 M sulfuric acid was added to a beaker with 50 g of dried sludge and was stirred continuously at 70 °C for 1 h. After aging the solution overnight and filtering, the ammonia solution with 10% concentration was added slowly until the pH of the solution reached pH 9 and the white precipitate was observed. The precipitate was washed with deionized water until a neutral pH was reached, then dried at 115 °C overnight. It was subsequently calcined in a furnace at 600 °C for 2 h, with a heating rate of 5°C/min, to obtain the extracted aluminum (EA). Subsequently, the EA was impregnated with a chromium precursor solution to achieve a 15 wt.% chromium metal loading, producing the material denoted as 15CE. The impregnation was conducted at room temperature and stirred at 250 rpm for 4 h. Finally, the sample was dried at 115 °C overnight and calcined at 600 °C for 5 h. The calcination conditions and metal ratio were selected based on reported works in the synthesis of CE catalysts [13].

The catalytic co-pyrolysis (CCP) of cotton fabric waste (CFW) and polypropylene waste (PPW) was conducted in a vertical fixed-bed reactor (Micromeritics). The reactor was loaded with 1 g of feedstock, consisting of a 1:1 CFW-to-PPW ratio and a catalyst in a 1:1 feedstock-to-catalyst ratio. Quartz wool was used to separate the feedstock and catalyst. During the CCP process, nitrogen gas (N_2) was introduced into the reactor to maintain an inert atmosphere, and the system was heated to a constant temperature of 500 °C. The vapors produced during the reaction were cooled in a condenser, and the final bio-oil liquid was collected from the condenser and wax trap. The main products, char, and bio-oil were weighed to determine the product yield. The percentage of yield and yield of gas were calculated using Eq. (1) and Eq. (2), respectively. The characterizations of bio-oil were performed by gas chromatography-mass spectrometry (GC-MS) using GC-MS-QP2010 Ultra, Shimadzu, and Fourier Transform Infrared (FTIR) analysis to determine the functional groups using a FTIR spectrum (PerkinElmer Frontier, United States) with spectrum range of 4000-6000 cm^{-1}. The selectivity and carbon number fraction yield were calculated using eq.3 and eq.4, respectively.

$$\text{Char and bio-oil yield, \%} = (\text{weight of product}/\text{weight of feedstock}) \times 100 \tag{1}$$

$$\text{Gas yield, \%} = 100 - \text{yield of char} - \text{yield of bio-oil} \tag{2}$$

$$\text{Selectivity, \%} = (\text{sum area of each compound} / \text{total area of all compounds}) \times 100 \tag{3}$$

$$\text{Carbon number fraction yield, \%} = (\text{sum area of each carbon number in hydrocarbon} / \text{total area of hydrocarbon}) \times 100 \tag{4}$$

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3. RESULT AND DISCUSSION

3.1 Influence of pyrolysis time on product yield

In this work, the influence of pyrolysis time was conducted between 15 min to 75 min at constant operating conditions and was counted after the temperature reached 500 °C. The evolution of CCP products as a time function is exhibited on Fig. 1. Varying the pyrolysis time significantly influenced the production of bio-oil and gas products. This was due to the separation of feedstock from the catalyst by ex-situ configuration, whereby the feedstock initially underwent thermal degradation to produce volatiles and flowed through the catalyst bed for secondary catalytic cracking to produce bio-oil.

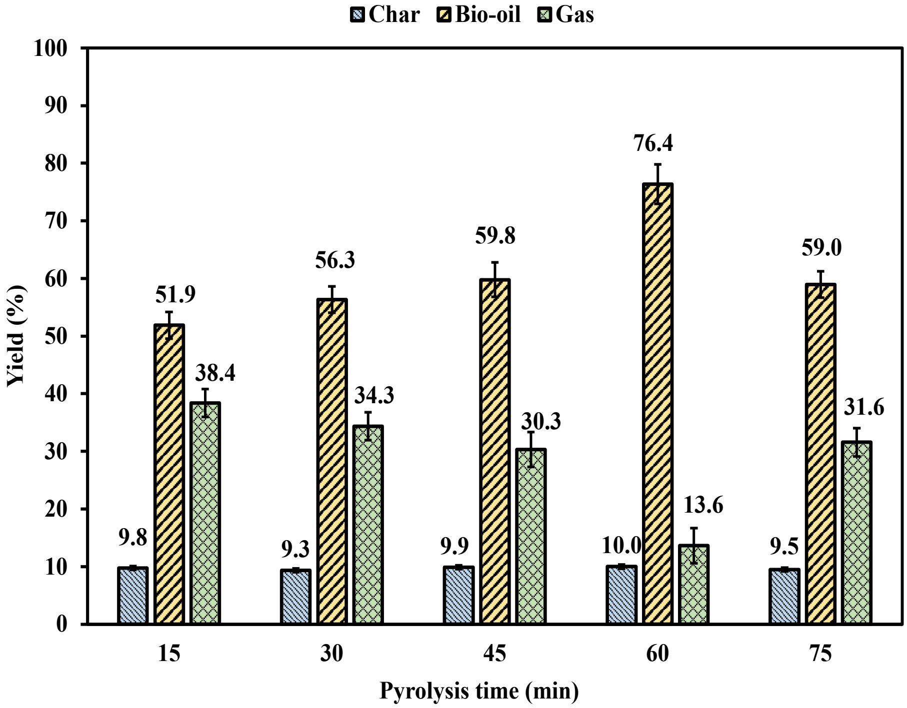

Fig. 1. Influence of pyrolysis time in catalytic co-pyrolysis of CFW and PPW (Temperature: 500 °C, CFW/PPW: 1:1, F/C: 1:1)

The residence time required for the decomposition of feedstock, such as biomass and plastic, should be longer than the vapor residence time to achieve higher yields and improved feedstock conversion [7]. As can be seen from Fig. 1, the increased pyrolysis time from 15 min to 60 min had increased the production of bio-oil yield from 51.9 % until it reached the highest of 76.4 %. A further increment in pyrolysis time showed a significant drop in bio-oil yield to 59 %. The extended duration facilitated cracking reactions, leading to the generation of smaller fragments and a higher quantity of liquid. Nevertheless, further increases in pyrolysis time beyond optimum time could enhance the gas yield. Conversely, incrementing the pyrolysis time displayed minimal effects on char yield, ranging from 9.3 % to 10 %. The devolatilization and thermal decomposition of the feedstocks occur rapidly during the initial pyrolysis stage. This indicates that the primary reactions responsible for converting the feedstocks into volatiles and gases are largely

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complete early in the process, leaving a relatively stable solid residue (char). At 500 °C, most of the PPW has entirely decomposed while CFW has reached its stable solid residue, as evidenced by the TGA results reported previously [13]. The presence of a catalyst might influence the composition and reactivity of volatiles, but it may not significantly impact the char once it has formed. Several studies have investigated the impact of pyrolysis time on product distribution. Maulinda et al., [14] reported that pyrolysis duration significantly affects bio-oil yield, with prolonged reaction times enhancing bio-oil production due to increased feedstock degradation. Similar observations were made by Ahmad et al. [12] and Huang et al., [15]. However, extending the pyrolysis time beyond an optimal point was found to increase gas production while substantially reducing char yield. Interestingly, further increases in reaction time eventually led to a stabilization of char yield [12]. Additional studies have shown that extended residence times promote secondary reactions such as carbonation, gasification, and thermal cracking, which ultimately reduce bio-oil yield [16-17].

3.2 Characteristics of bio-oil samples

The chemical composition of bio-oil products during catalytic co-pyrolysis of CFW and PPW was obtained via the GC-MS technique. The major compounds were classified into several groups, namely, hydrocarbons (HC), aromatic hydrocarbons (aromatic HC), alcohol, phenols, furans, carbonyl, acid, and sugar. The selectivity of the compounds is displayed in Fig. 2. In the CCP process, the main produced compound was found to be HC, followed by alcohol, furans, and carbonyl, while acids and sugars were reduced. As the pyrolysis time increases, the production of hydrocarbons has increased from 15 min to 45 min. However, the catalytic reaction favors the formation of oxygenates such as alcohol, phenols, furans and carbonyl when the pyrolysis time was prolonged to 60 min. The high formation of carbonyls and alcohols might be attributed to retro-aldol condensation of sugar derived from cellulose degradation [18]. Furthermore, insignificant formations of aromatic HC were observed from 15 min to 60 min, which was ascribed to a high energy barrier for cyclization and deoxygenation [19]. With the excess formation of the furans and carbonyls at 60 min, it had enhanced the formation of phenols [13]. On the other hand, a dramatic shift occurs in the chemical composition observed at 75 min of pyrolysis time, with a drop in phenols, furans, and carbonyls and the appearance of aromatics. The formation of aromatic HC can be formed in many pathways, such as via the Diels-Alder reaction, hydrocarbon pool, cyclization, and aromatization [16–17]. In the study by Maulinda et al. [14], phenol was identified as the predominant compound in the bio-oil derived from the pyrolysis of *Mimusops elengi*, accounting for 64.2% of the composition. This high phenol content is attributed to the extended reaction time, which facilitates more effective lignin decomposition into hydrocarbon compounds. In contrast, Tsai et al. [22] investigated the effect of residence time, varying it from 1 to 8 min during the pyrolysis of rice husk. Their results indicated that aromatic and carbonyl compounds were consistently dominant across all residence times.

The result in Fig. 3 shows liquid products consisting mainly of aliphatic alkene and cycloalkene. Besides that, it also consists of aliphatic alkane, a small fraction of cycloalkene, and aromatic hydrocarbons. As polypropylene plastic is used in the reaction, the aliphatic alkene is prone to produce more in bio-oil [1], and the result displays a slight influence of time in hydrocarbon compositions. On the other hand, the carbon numbers yield in Fig.4 shows the significant formation of gasoline (C8-C12) and diesel (C13-C20) fuel range with major components of hydrocarbons in bio-oil in the diesel range. There are also formed waxy range fuels (>C20); nonetheless, it is insignificant. The lighter carbon chain of C8-C12 rose slightly, while the heavier carbon chain (C13-C20) was reduced by time. This is exceptional for the hydrocarbons produced at 60 min, which conversely produce more gasoline range hydrocarbons. The complex interactions between CFW-PPW and 15CE catalyst, in some cases, for instance, at 60 min, caused different characteristics of the oil.

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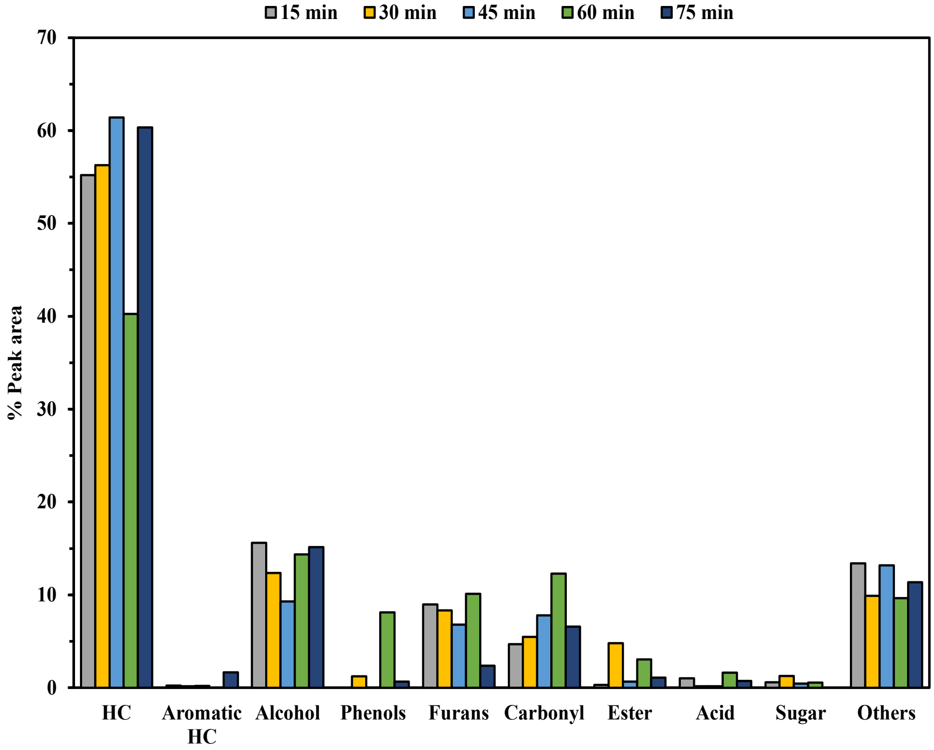

Fig. 2. Bio-oil chemical composition at different pyrolysis times (Temperature: 500 °C, CFW/PPW: 1:1, F/C: 1:1)

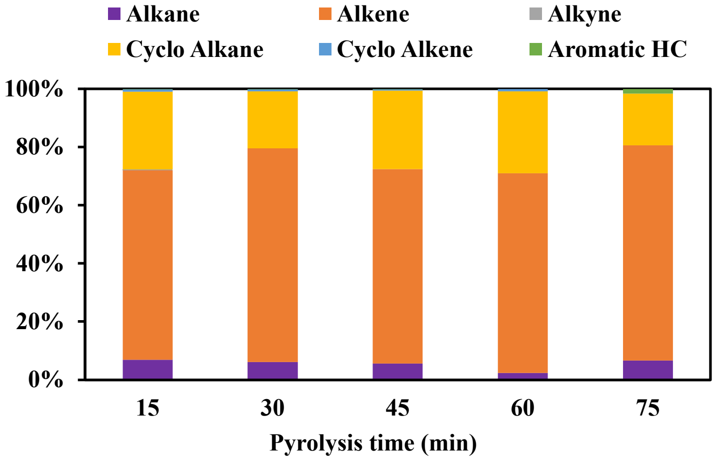

Fig. 3. Hydrocarbon selectivity at different pyrolysis times (Temperature: 500 °C, CFW/PPW: 1:1, F/C: 1:1)

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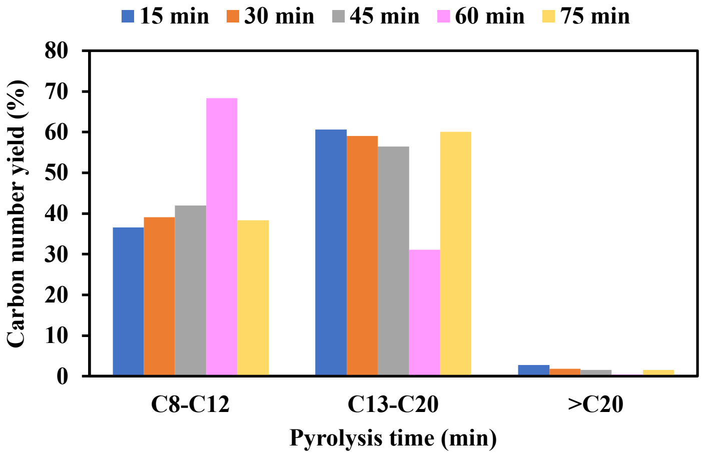

Fig. 4. Carbon number fraction yield (%) at different pyrolysis times (Temperature: 500 °C, CFW/PPW: 1:1, F/C: 1:1)

3.3 FTIR analysis of bio-oil samples

FTIR studies have identified characteristic absorption bands that correlate with various functional groups present in the bio-oil. Fig.5 displays the FTIR spectrum from 600 cm^{-1} to 4000 cm^{-1} at different pyrolysis times. In general, the spectrum shows similar patterns in wavelengths except for minor differences in intensities.

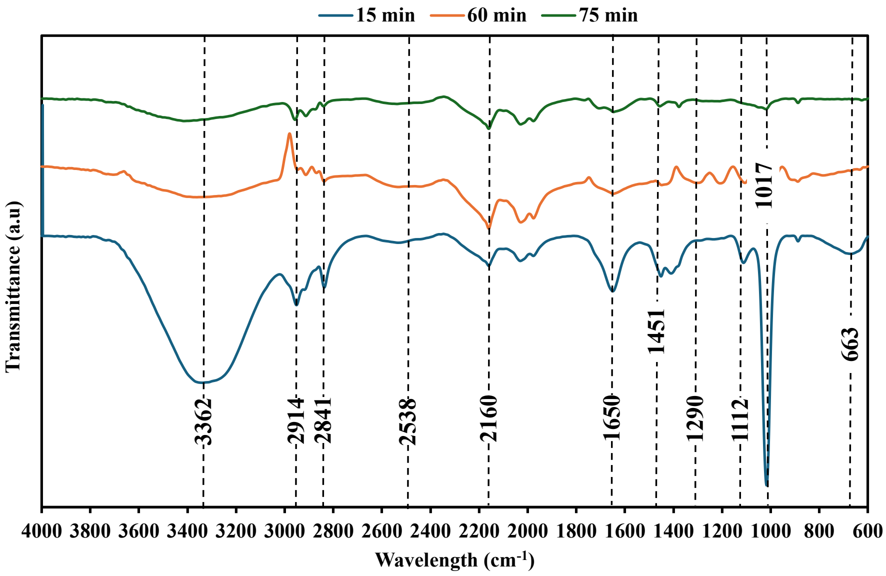

Fig. 5. FTIR spectrums of bio-oil at different pyrolysis times (Temperature: 500 °C, CFW/PPW: 1:1, F/C: 1:1)

The C-H stretching vibrations, in the range of 2800-3000 cm^{-1}, and C=C stretching at the range 1630-1670 cm^{-1}, commonly observed in biomass and plastic pyrolysis, indicate aliphatic hydrocarbons [23]. The

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broad spectrum of O-H stretching at the range of 3200-3500 cm $^{cm^{-1}}$ and sharp spectrum at 2914 cm^{-1} indicates the presence of alcohol compounds in bio-oil. Additionally, the presence of other oxygenate compounds such as ether (1112 cm^{-1}), ketone (1705 cm^{-1}), carboxylic acids/aldehyde (2841 cm^{-1}), ester/anhydrides (1112 cm^{-1} and 1290 cm^{-1}) and amides (1650 cm^{-1}) were also identified. On the other hand, the spectrum also displayed the presence of inorganic compounds, such as from the halide group at the fingerprint region (<1500 cm^{-1}). Obviously, the diminished ester (C-O) and halides (C-X) functional group has longer pyrolysis times as the intensity weakens, as shown at spectrum 1017 cm^{-1} and 663 cm^{-1}, respectively. Other than that, peaks indicate phenols and aromatic usually appear at broad O-H stretching (3200-3600 cm^{-1}), which overlapped with the alcohol spectrum. Similarly, the aromatics peak is found to overlap with the C=C of alkene (1450-1600 cm^{-1}).

4. CONCLUSION

The influence of pyrolysis time in catalytic co-pyrolysis (CCP) of CFW and PPW over 15CE catalysts was studied. The result demonstrated that the pyrolysis time significantly impacts product distribution, especially bio-oil. In general, the catalytic reaction enhanced the production of hydrocarbon, reduced acid, and converted sugar into valuable fuel and chemical products. Increased pyrolysis time resulted in an increase in bio-oil yield, and the conversion of CFW-PPW to bio-oil was as high as 76.4 % in 60 min. GC-MS result proved that varying pyrolysis times during the CCP process might change the bio-oil composition with most of the product produced HC. The oxygenate compound, especially alcohols and intermediates (phenols, furans, and carbonyls), were formed significantly at 60 min. Nevertheless, the yield of bio-oil and intermediate compound was reduced when the pyrolysis reaction time was prolonged to 75 min. Most of the HC produced aliphatic alkene and cyclo-alkane with the carbon number yield at the diesel range. The presence of chemical compounds in bio-oil was confirmed with the FTIR spectrum. The thermal decomposition of the feedstocks occurs rapidly during the initial stage of pyrolysis, making the volatiles and gases complete early in the process as soon as 15 min of pyrolysis time. Thus, the pyrolysis time is prolonged, leading to a secondary reaction and changing the chemical composition of the bio-oil.

5. ACKNOWLEDGEMENTS

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6. CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefits or commercial or financial conflicts and declare the absence of conflicting interests with the funders.

7. AUTHORS' CONTRIBUTIONS

Nur Alwani Ali Bashah: Conceptualization, methodology, formal analysis, investigation, and writing-original draft; **Wan Zuraida Wan Kamis**: Conceptualization, supervision, writing- review and editing, and validation; **Muhammad Zahiruddin Ramli**: Conceptualization, supervision, writing- review and editing, and validation; **Siti Shawalliah Idris**: Review, and validation; **Mohamad Anuar Kamaruddin**: Review, and validation.

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