

Effect of Freeze-Drying Pre-treatment on the Properties of Activated Carbon Derived from Arabica Coffee Pulp

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Abstract

Activated carbon is a highly valuable material due to its large surface area. Even though coffee pulp has a high fixed carbon content, research on its utilization as a raw material for activated carbon production remains very limited. This study investigates the impact of freeze-drying as a pre-treatment method on the properties of activated carbon derived from coffee pulp waste. The goal is to enhance its potential application as an energy storage material. The research methodology includes: (i) washing and soaking of coffee pulp waste as the raw material, (ii) freeze-drying, (iii) pyrolysis, and (iv) activation. Activation was carried out using three different methods: chemical activation with KOH, physical activation with nitrogen at 800°C, and a combination of both methods. Characterization results indicate that freeze-dried samples exhibit improved thermal resistance and porosity. Furthermore, the functional group analysis confirms the removal of undesirable compounds. These findings demonstrate that freeze-drying pre-treatment significantly influences the properties of activated carbon derived from coffee pulp waste, highlighting its promising potential for energy storage applications.

Keywords: activated carbon; coffee pulp; freeze drying; KOH

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INTRODUCTION

Activated carbon is a porous material that has a high surface area and a large micropore volume (Ganjoo, Sharma and Kumar, 2023). Activated carbon is widely used in various applications, especially in the industrial sector. For example, it is utilized as an

absorbent for purifying food and drink ingredients in the food and beverage industry, and removing unpleasant odor of domestic waste water. Activated carbon can also play significant role as a catalyst support in the chemical reactions as well as an

immobilizing agent for enzyme and microorganisms in biochemical reactions.

A careful selection of raw material for activated carbon preparation is one of the strategies to obtain good properties. Lignocellulosic biomass is one of the raw materials that can be used as a porous activated carbon material because it has a high fixed carbon value. The recent increase in coffee demand making coffee pulp is abundantly available in Indonesia, especially in Aceh Province. So far, this agricultural waste is usually disposed to the environment by the local people because they lack of knowledge. If this situation is allowed to continue, it can trigger environmental problem and endanger human health because this waste also contains carcinogenic substances. Coffee pulp waste is one of biomass residues that has a promising potential for use as a raw material for activated carbon production. Sulhatun *et al.* (2023) reported that coffee pulp contains 10,8% ash, 58,2% volatile matter, 8% moisture, and 23% fixed carbon. In addition, it also bears 12% hemicellulose, 42% cellulose, and 2% lignin. Meanwhile, Setiawan *et al.* (2020) found that coffee pulp contains about 62% fixed carbon. The serious drawbacks that limits the use of coffee pulp as a raw material for activated carbon preparation are its low-density value and soft structure.

Process of making activated carbon involves at least three main stages, that are pre-treatment of raw materials, carbonization, and activation (Gan, 2021). The pre-treatment process for raw materials can be carried out in various ways, namely by washing/cleaning, soaking, grinding, drying, and others. During the washing and soaking process, the easily soluble contaminants will be removed. Taer *et al.* (2021) claimed that the washing and soaking processes can reduce the ash content from 19.79% to 11.62% as the result of alkali and alkaline earth metals removal (Singh Karam *et al.*, 2022). In addition, Abulikemu *et al.* (2023) reported that size reduction of raw material via grinding process is capable of producing activated carbon with a fairly high surface area (1107 m²/g). In fact, during grinding process, more abrasion takes place on the samples, resulting in the formation of pores. Furthermore, the drying process of raw materials either through conventional oven drying or freeze drying also greatly influences the active carbon produced. Drying using an oven is quite common and is often used by researchers because the process and costs are not too expensive. However, there is something that is a challenge to this process, namely that it can damage the structure of the raw material, resulting in poor results for very soft raw materials. This can damage the structure of the raw material when processed further. Based on this, the drying process can be done using a freeze-dryer.

The freeze-drying method is believed to provide better results because this process removes water in the material tissue slowly using the sublimation method. This process can maintain a better pore structure compared to conventional drying

methods. Until now, as far as literature searches have been carried out, the use of the freeze-drying method for the drying process in producing activated carbon is still very limited. This drying method is able to produce high efficiency in removing water from the plant matrix and is able to keep all bioactive components stable. This method is often the method of choice for extracting flavonoids (Jurinjak Tušek *et al.*, 2022). Apart from that, the freeze-drying method also has a big influence on various properties of the final product, including the presence and composition of bioactive components such as active carbon and the physicochemical and organoleptic properties (Belwal *et al.*, 2022). The results obtained from this pre-treatment can also be influenced by the carbonization process carried out.

The carbonization process is a stage in the process of making active carbon by heating organic materials with little or no oxygen gas contained in the reactor (Basu, 2013). During the carbonization process, many parameters must be considered, one of which is the temperature. If the temperature used is too high, the biochar yield will be low. Qurat-ul-Ain *et al.* (2021) revealed that the pyrolysis of *Parthenium hysterophorus* at 350°C and 600°C resulted in a decrease in the biochar yield value from 61%-37%. Temperature also affects the quantity of fixed carbon produced. Setiawan *et al.* (2024) reported that pyrolysis of Gayo Arabica coffee-pulp at 400°C obtained biochar with a fixed carbon content of 55%. Temperature also affects the quantity of fixed carbon produced. However, if the temperature is very high an excessive biochar cracking can occur leading to the formation of many macropores, which is unacceptable for use as an energy storage material. In addition, if the pyrolysis temperature is too low also will not generate good results because it cannot open the pores optimally. The further strategy that can be carried out to obtain mesopores is by the activation process (Farma *et al.*, 2021).

Activation processes are divided into three methods, namely the physical, chemical and chemical-physical. An important thing to note is that the use of the type of activator greatly influences the quality of the activated carbon produced, especially the surface area and pore volume. Each type of activator has a different effect on the pore structure of activated carbon, so selecting the right method is very important for the specific application (Raihan *et al.*, 2020). Raw materials that are classified as hard should be physical activation at high temperatures so that more pores are opened. Previous research by Shahcheragh *et al.* (2023) utilized palm kernel as a raw material and employed physical activation at 600°C for three hours to synthesize activated carbon. The SEM images of the resulting products showed that many pores were visible with a more uniform size suggesting its potential for use as an energy storage material. However, for raw materials that are relatively soft, chemical activation can be a better option. Yanti *et al.* (2024) used coffee pulp as a raw material and chemical

activation using 1% w/v KOH solution to produce activated carbon. The result obtained was a iodine absorption capacity of 545.67 mg/g.

Research on activated carbon continues to develop and many new methods can be discovered by varying the raw materials used. Recent research shows that through the pyrolysis method at 400°C with chemical activation using 10% ZnCl₂ solution, a surface area of 164.1 m²/g was attained (Nurmalita *et al.*, 2022). However, this surface area value is still relatively low for use as an energy storage material. Therefore, many efforts must be performed to achieve the desired surface area value. One of the strategies is by adjusting the pre-treatment of raw materials, such as drying methods. Earlier, Li *et al.* (2022) used a drying method using a freeze dryer at a temperature of -40 C for 12 hours followed by carbonization at 400°C for one hour and chemical activation using KOH and physical activation at a temperature of 800°C to obtain a fairly high surface area, namely 1300.47 m²/g. Based on this information, the coffee pulp waste which will be used as a raw material in activated carbon preparation should undergo appropriate pretreatment to obtain better properties.

In this research, the drying method for the pre-treatment of the coffee pulp waste was carried out using a freeze dryer. The aim is to study the effect of drying methods on the physical and chemical characteristics of active carbon derived rom coffee pulp waste, which can be used as a reference for further scale up and implentation in energy storage system and other possible applications.

MATERIALS AND METHODS

Materials

Arabica coffee pulp as raw material was collected from a coffee agroindustry in Bener Meriah, Aceh, Indonesia. Tap water and nitrocellulose (NC) thinner used for washing and soaking were purchased from local chemicals store in Meriah, Aceh, Indonesia. Potassioun hydroxide (KOH) supplied by Merck KGaA, Darmstadt, Germany was used for chemical activation. Nitrogen gas (99.99% v/v purity) was sourced from PT. Samator Indo Gas Tbk. was used for physical activation.

Methods

Initially, the raw material was washed and soaked in tap water for one night and followed by washing with nitrocellulose (NC) thinner to remove dirt and ash contained in the raw material. Then the samples were drained and dried under the sun for eight hours, before further dried using a freeze dryer at -40°C for 24 hours. The dried sample were then subjected to a pyrolysis process using the equipment set up previously used by Setiawan *et al.* (2024) at 480°C for one hour. The biochar yield was calculated using the equation (1).

$$\text{Biochar Yield} = \frac{\text{mass o biochar (g)}}{\text{initial mass of feedstock (g)}} \times 100 \dots (1)$$

The biochar obtained from pyrolysis process was then subjected to chemical, physical and chemical-physical activation processes. Chemical activation was performed using 2 % w/v KOH solution, while the physical activation was conducted by heating at 800°C for 30 minutes under nitrogen environment.

Characterization

The activated carbon obtained from coffee pulp was characterized by carrying out TG (Thermogravimetry), Scanning Electron Microscopy (SEM) and Fourier Transform Infra Red (FTIR) Spectrophotometry analysis. The thermogravimetric analysis uses a Shimadzu DTG-60 machine by flowing nitrogen at a flow rate of 20 mL/min and heated at a heating rate of 10°C/min. All samples were placed in an alumina crucible and heated to 600°C. Data on changes in mass and temperature was read and recorded directly in a computer system, then graphs can be plotted using the Origin Lab application.

Table 1. Sample code description

Sample code	Description
CPOV	Oven-dried coffee pulp
CPFD	Freeze-dried coffee pulp
FBCCP	Biochar produced from freeze-dried coffee pulp
FACKOH	Activated carbon prepared from freeze-dried coffee pulp and activated by KOH solution
FAC-800	Activated carbon produced from freeze-dried coffee pulp and activated by heating at 800°C under N ₂ environment.
FACKOH-800	Activated carbon produced from freeze-dried coffee pulp and activated by KOH solution followed by heating at 800°C under N ₂ environment.

The analysis of surface morphology of the activated carbon was carried out using a back-starred electron detector (BSE) of JEOL JSM-6510 Series Scanning Electron Microscope. The method used in the analysis is to freeze cellulose-activated carbon powder on aluminum until dry. Then the sample was sprinkled with gold for 30 seconds using a polaron and the results were displayed in stereoscan. The functional groups of the activated carbon was carried out using an FTIR employing the Shimadzu IR Prestige 21, where 0.2 mg of active carbon was mixed with 2 mg of KBr and formed into pellets. Then this pellet is fed to an FTIR instrument with a wave number of 5000-400 cm⁻¹. The products obtained from this research were coded to enable their easy recognition as shows in Table 1.

RESULTS AND DISCUSSION

Pyrolysis of Coffee Pulp

The carbonization process of coffee pulp was conducted for both types of feedstock, namely the oven-dried coffee pulp (CPOV) and freeze-dried coffee pulp (CPFD). Figure 1 presents the temperature variations recorded during the carbonization process. This process was carried out in a pilot-scale reactor heated with the help of an LPG burner, as previously reported by Setiawan *et al.* (2024). According to the graph, both CPOV and CPFD experiments reached a pyrolysis reactor temperature of 480°C within 50 minutes, indicating a heating rate of 9°C/min classifying the process as a slow pyrolysis. The results confirm that the pyrolysis behavior of the feedstock remained consistent regardless of the drying method, showing no significant effect of the drying technique used as the pre-treatment.

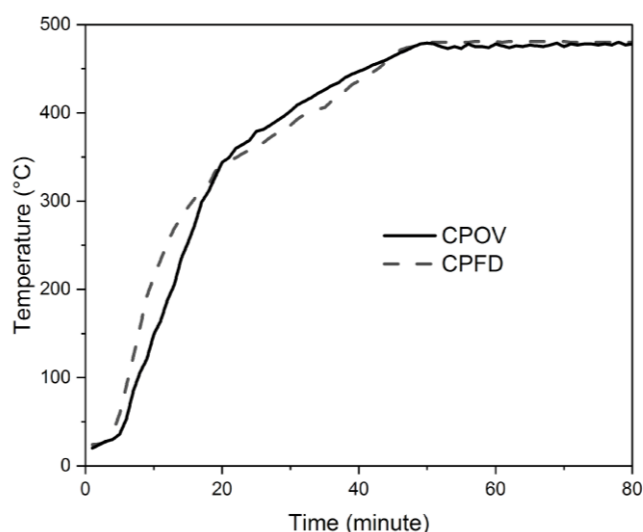


Figure 1. Temperature profile pyrolysis of coffee pulp obtained from two different drying techniques

The biochar yield from the pyrolysis process obtained by each type of sample was different, where the CPOV and CPFD samples achieved 45% and 44% yield, respectively. This difference is most likely to be influenced by the microstructure of the raw materials fed into the pyrolysis reactor. This result is in accordance with previous research (Meng and Wang, 2020a), which the pyrolysis obtained 45% and 36% biochar yield when the pyrolysis reactor was fed by oven-dried and freeze-dryer biomass. This is because biomass treated with freeze drying forms a more uniform pore structure compared to biomass dried using a conventional oven. The freeze-drying method is efficiently helpful in converting the volatile matter components, such as renin acid, furan, esters, and cyclopentene into gas and further releases them from the biomass matrices (Karimi and Taherzadeh, 2016; Gao *et al.*, 2017).

Thermogravimetry Analysis

The results of the thermogravimetric analysis (TGA) of the four samples are shown in Figure 2. In general, the TGA plots show good the thermal stability of the activated samples. These plots also help in providing insight into deactivation of activated carbon in terms of weight loss during heating under N₂ flow. The different thermal events found in each plot, mostly related to moisture loss, volatile release, and carbon burnout. Four different TGA curves depicted in Figure 2 were obtained from freeze-dried samples, where all physically activated samples exhibited better thermal stability than those without physical activation. This is due to the influence of the physical activation temperature performed at 800°C, by which more organic substances were decomposed and resulted in an increase in fixed carbon content. This suggests that this activated carbon can potentially be used as supercapacitor material, since it has good thermal stability and porosity. Biomass-derived activated carbons have been shown as electrode materials for supercapacitors (SCs) due to their renewability, low-cost, and ready availability. (Ahmad *et al.*, 2023).

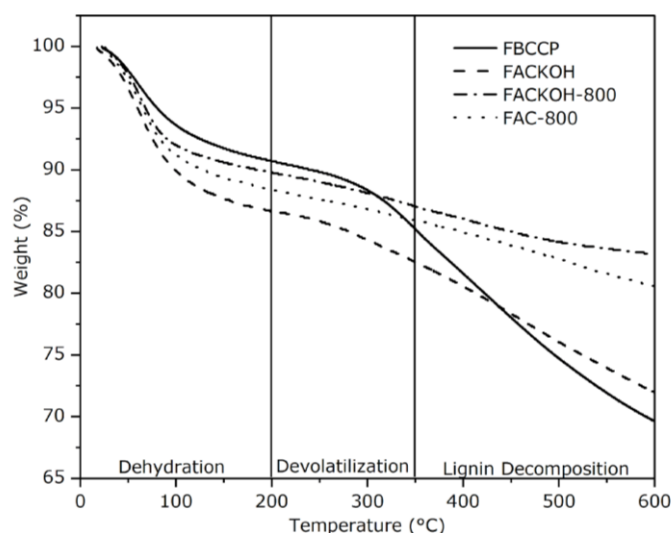


Figure 2. Thermograms of Biochar samples at heating rate 10°C/min and flow rate nitrogen of 20 mL/min.

Figure 2 shows that the FBCCP sample lost 30% mass. This suggest that more samples were lost in samples without activation because they were only pyrolyzed at a temperature of 480°C, as a result, there were still many substances in the samples that yet to decompose into liquid or gas. In contrast, the FACKOH sample lost about 28% mass, which is likely due to the additional influence of nitrocellulose thinner during pre-treatment. Nitrocellulose thinner can add volatile matter during chemical activation, because it contains organic solvent, mainly ethyl acetate (Xu *et al.*, 2020). Due to its high vapor pressure, this substance will transform into gas during heating and react with activated carbon. Furthermore, the

FACKOH-800 sample lost 19% mass. Both FACKOH sample and FACKOH-800 have a greater percentage of mass loss due to the effect of immersion in nitrocellulose thinner. Lastly, the FAC-800 sample demonstrated 17% mass loss. The difference in mass loss values was not much because the physical activation employs quite high temperature (800°C) that allows better vaporization of volatile compounds (Borghei *et al.*, 2021). Based on the TGA results of the activated samples, mass loss was not significant.

In general, the TG graphs of the four samples show that the mass decomposition occurred in three stages, namely dehydration, devolatilization, and lignin decomposition (Herak, 2021). These three stages are interconnected and have different temperature characteristics. The dehydration stages occur in each sample with a different amount of mass reduction and temperature, starting from room temperature to 200°C. The FBCCP sample experienced a mass decrease of 2% at 41°C (onset), and then it experienced a mass decrease of 9% at 161°C (endset). The FACKOH sample experienced a mass decrease of 3% at 46°C (onset), and then it experienced a mass decrease of 10% at 97°C (endset). Meanwhile, the mass decrease experienced by FACKOH-800 sample at 47°C (onset) and 89°C (endset) were 3% and 9%, respectively. Finally, the FAC-800 sample experienced a mass decrease of 3% and 8% at 48°C (onset) and 98°C (endset), respectively. Physically, the decrease in sample mass during TG analysis is due to the loss of water and other volatile compound molecules contained in the biomass pores. However, Figure 2 also shows that each sample still contains water.

Depending on the type of sample, the devolatilization stage generally takes place in a temperature range of 200-350°C with a decrease in sample mass. This stage occurs due to the devolatilization process of the sample's components like the carbohydrates, lipids, several proteins and lignin (Nurmalita *et al.*, 2022).

Finally, the third stage happens in a temperature range of 350-600°C, where not much material is lost because it is dominated by lignin degradation and can withstand high temperatures (Apaydin Varol and Mutlu, 2023). The results of this process also contain several inorganic compounds called carbon solids (Herak, 2021). Based on these results, it can be observed that the freeze-drying pre-treatment process affects the thermal stability of the sample because freeze-drying produces a neater and more orderly structure. Samples that have a neater structure make the decomposition process easier (Shahcheragh *et al.*, 2023). However, the activation process can also influence the thermal stability of a sample.

SEM Analysis

The scanning electron microscopy (SEM) analysis was carried out to observe the morphological structure of the sample. The SEM analysis of activated

carbon was conducted at the same magnification of 2500× and the results are depicted in Figure 3. Figure 3 displays that each sample has a different surface morphology.

Figure 3 shows that the FBCCP sample still has very small and nearly invisible pores because the sample has not been activated. This result is in good agreement with Raihan *et al.*, (2020) who pyrolyzed Robusta coffee peel at 400°C, which resulted in almost invisible pores. As a result, the biochar was not able to absorb other ingredients optimally because it still mixed with other impurities and no activation was conducted, such as potassium (Raihan *et al.*, 2020).

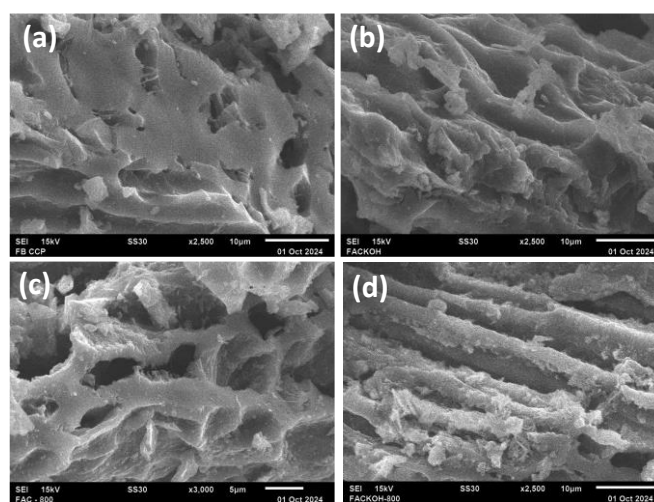


Figure 3. Morphology sample; (a) FBCCP, (b) FACKOH, (c) FAC-800, (d) FACKOH-800.

As seen in Figure 3, the FACKOH sample has more visible pores, because of the influence of nitrocellulose thinner during washing, which helps in reducing the impurities and other unwanted components in the biochar. Previous studies reported that the main impurities include potassium (K), chlorine (Cl), and zinc (Zn) (Nurmalita *et al.*, 2022). Additionally, the formation of pores in both samples could be attributed to the role of the KOH as the activator, which facilitates the breakdown of lignocellulose and leaves more new pores in the activated carbon (Espinoza-Acosta *et al.*, 2018). In addition, the chemical activation applied to the sample also reduced potassium levels, thereby increasing the presence of pores and enhancing absorption capacity of the resulting activated carbon (Raihan *et al.*, 2020). However, it should also be noted that the use of high activator concentration and lengthy chemical impregnation during activation step can lead excessive dehydration of carbon, which can erode the pore walls and cause the pore framework to collapse.

The FAC-800 sample exhibited more open pores than the other samples as a result of better lignin and other volatiles removal from the sample activated by high temperatures, reaching 800°C (Lu *et al.*, 2017). A higher activation temperature allows the activator solution to penetrate into the inner tissue of

the sample, react with the residing substance, and ultimately produce more pores (Shahcheragh *et al.*, 2023). This result is in good accordance with research (Li *et al.*, 2022), which claimed that samples that were physically activated at 800°C produce more intact pores. This result suggests that volatile matter substances are easily released from the freeze-dried samples.

Finally, the FACKOH-800 sample exhibits a unique surface structure, which is characterized by the appearance of long-straight channels due to the detachment of soluble substances as a result of the addition of nitrocellulose thinner during washing of the raw material. Besides, it can also be due to the influence of lengthy chemical activation using KOH solution and thermal activation at high temperatures. Indeed, the resulting pores become more visible because freeze-drying pre-treatment can improve the pore structure of the biochar (Meng and Wang, 2020b).

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR analysis is commonly performed to identify the presence of specific functional groups in the biochar before and after activation (Sun *et al.*, 2024). In this research, the samples were subjected to FTIR analysis at a wave number range of 4000-500 cm^{-1} . The analysis results are divided into four sections, namely the single bond, double bond, triple bond, and fingerprint sections. Based on the FTIR results presented in Figure 4, it is clear that the samples experienced functional groups changes. Samples dried using a freeze dryer demonstrated fewer functional groups compared to those dried using a conventional oven (Meng and Wang, 2020b). Presumably, the use of nitrocellulose thinner for soaking of the coffee caused this difference because nitrocellulose thinner has the ability to remove impurities from the coffee pulp waste.

In the single bond section, a compound is detected by the appearance of a peak in the band range of 3490 – 3280 cm^{-1} . Freeze-dried samples still contained a lower amount of caffeine. This is due to the use of nitrocellulose thinner that can modify the sample structure leading to reducing the number of functional groups. This observation is similar to that reported by Nurmalita *et al.* (2022) who found caffeine in their coffee pulp waste sample, but at a different wave number, namely 2941-2829 cm^{-1} .

Two peaks appeared in the triple bond section with a wave number range of 2500-2000 cm^{-1} , namely at 2349.3 cm^{-1} and 2341.58 cm^{-1} for FACKOH-800 and FBCCP, respectively. These wave numbers indicate the presence of N-H bond, which possesses the ability to increase both the hydrophilicity and wettability of the activated carbon. From the energy storage point of view, this bonding affects the increase in specific surface area and electrochemical

performance of activated carbon, specifically in the supercapacitor applications (Li *et al.*, 2018).

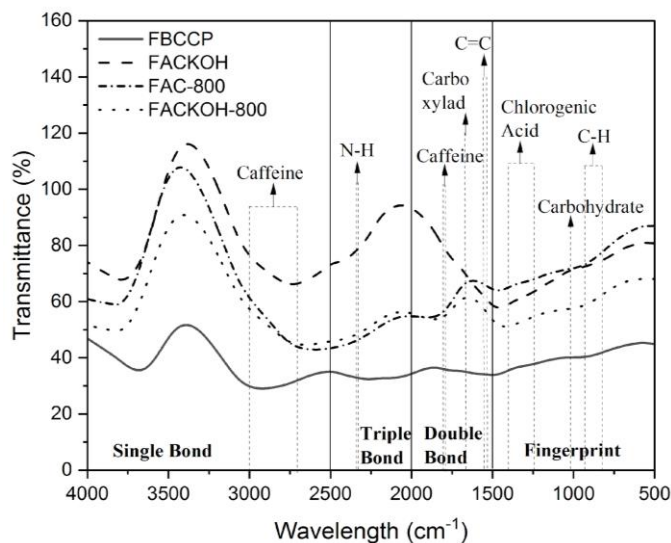


Figure 4. FTIR spectra of sample activated carbon

In the double bond section, two compounds were identified with wave numbers ranging from 2000-1750 cm^{-1} . The identified compounds contain caffeine but at different wave numbers, which were respectively 1793.8 cm^{-1} and 1797.66 cm^{-1} in the FACKOH-800 and ACKOH-800 samples. In addition, carboxylic groups were confirmed at wave number of 1685.79 cm^{-1} in the FBCCP sample. Furthermore, wave numbers of 1510.26 cm^{-1} , 1519.91 cm^{-1} , and 1504.48 cm^{-1} were assigned for the presence of C=C bonds in the FBCCP, FAC-800, and FACKOH-800 samples, respectively. However, no important functional groups were detected in the FACKOH sample, most probably due to the influence of KOH solution used in the chemical activation process that completely cleans up the sample structure.

In the final section, which is in the fingerprint, samples dried using a freeze dryer contain chlorogenic acid and carbohydrate compounds with different wave number. The same results were reported by Nurmalita *et al.* (2022) where they detected the presence of chlorogenic acid at a wave number of 1404-1242 cm^{-1} . In this research, the FBCCP samples that were dried using a freeze dryer exhibited the presence of chlorogenic acid, which was identified at a wave number of 1211.3 cm^{-1} . The FAC-800 sample exhibited the presence of chlorogenic acid based on the peaks at wave numbers of 1220.94 cm^{-1} and 1338.6 cm^{-1} . Meanwhile, the peaks at wave number of 1053.13 cm^{-1} , 1211.3 cm^{-1} , 1346.31 cm^{-1} , 1425.4 cm^{-1} , and 1296.16 cm^{-1} can be assigned as the indicators for the existence of chlorogenic acid in FACKOH sample. Finally, the C-H bond was identified in the FACKOH and FBCCP samples at wave numbers of 825.53 cm^{-1} and 926.9 cm^{-1} .

The FTIR results confirmed a reduction in functional groups in the activated carbon obtained due to the effect of drying using a freeze dryer, which is in

accordance with the previous research (Meng and Wang, 2020b) This finding also reveals that the use of freeze-drying for the pre-treatment of Arabica coffee pulp can alter its chemical composition. Moreover, the activation process also potentially affects the chemical composition of the active carbon produced.

CONCLUSION

The effect of freeze-drying on the Arabica coffee pulp as the pretreatment for preparing activated carbon has been investigated. The freeze-drying pretreatment improves the thermal stability of the resulting activated carbon. Furthermore, the activated carbon obtained possesses a large number of pores in its internal structure with a remarkable reduction of the undesirable functional groups suggesting its promising potential for use as an energy storage material.

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