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Research Paper

Study and Characterization of Hydrochar from Duku (*Lansium domesticum*) Peel

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Abstract
Peel of duku fruit (*Lansium domesticum*) was prepared into hydrochar by using the hydrothermal carbonation method at heating time variations of 8, 10, 12, and 24 hours and temperature variations of 200 and 250°C. X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FT-IR), Brunauer Emmet Teller (BET), and Scanning Electron Microscope (SEM) analyses were conducted to determine at what time and temperature variations the adsorbent had the best adsorption quality. Based on the characteristics of the adsorbent, it can be seen that the best hydrochar is at 12 hours and a temperature of 200°C. X-Ray Diffraction (XRD) analysis showed the presence of diffraction peaks at angles of 15.7° and 22.79° Fourier Transform Infra Red (FT-IR) analysis obtained explained that there were peaks of vibration peaks namely -OH, -CH, =CH, C=O, C=C aromatic and aliphatic. Brunauer Emmet Teller (BET) analysis can be seen that the increase in surface area on duku fruit peel (*Lansium domesticum*) and hydrochar at 200°C from 12.343 m^2/g to 22.635 m^2/g. Scanning Electron Microscope (SEM) analysis shows that the surface peel of duku fruit (*Lansium domesticum*) material has a clumped surface morphology in the same phase or also called aggregation, while the hydrochar tends to have an irregular shape or can be called heterogeneous morphology.

Keywords
Lansium domesticum, Carbonization, Hydrochar

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1. INTRODUCTION

Duku (*Lansium domesticum*) is a tropical plant native to Malaysia and Indonesia (East Kalimantan). Peel of duku fruit (*Lansium domesticum*) contains seco-onoceranoids, which is one type of triterpenoid in the form of lanceolate acid (bicyclic triterpenoid) and lansiolic acid. Duku fruit peels also contain lansiosides A, B, and C, which are examples of new structures of triterpenoid glycosides-amino sugars (Salim et al., 2016). Another content of duku fruit peel is 3-oxo-αbourbonene, including sesquiterpenoids which are the staple element of volatile compounds consisting of three isoprene units or fifteen carbon atoms. Various organic compounds such as triterpenoids, cellulose, and lignin that will increase their ability to absorb dye contaminants. The utilization of fruit peel waste as an adsorbent can help address various environmental issues such as wastewater pollution and agricultural waste disposal (Lam et al., 2016).

Hydrothermal carbonization is a wet thermochemical method that uses water as a medium using relatively low temperatures to obtain carbon materials (Triyono et al., 2016; Hussin et al., 2023). There are many methods used to produce carbon materials such as biofuels, briquettes, biochar, hydrochar, and others (Huseini et al., 2018; Sennou et al., 2020). The difference in the material produced depends on the type of method, the temperature used during the process, and the usefulness and stability of the resulting structure (Pauline and Joseph, 2020). Hydrothermal carbonization is the most promising method to convert biomass into value-added products. Hydrothermal carbonization is a very potential technique to be developed in the manufacture of pollutant-absorbing materials. This is because hydrothermal carbonization is carried out at relatively low temperatures, using water as the carbonization medium, and can produce materials rich in oxygen-containing functional groups (Yu et al., 2022; Juleanti et al., 2022).

One of the solid products produced from the hydrothermal carbonization method is hydrochar (HC) which is produced in the temperature range of 170-280°C with the resulting pressure of 2-5 MPa (Khoshbouy et al., 2019). Hydrochar can be utilized for various purposes, one of which can be used as a pollutant absorber (Islam et al., 2017). Hydrochar is the main product of the hydrothermal carbonization process, which is carbonaceous organic material. Hydrochar has a chemical composition, which is similar to coal at the lowest level such as peat or lignite (Kantakanit et al., 2018; Mlonka-Mędrala et al., 2022). Hydrochar has many uses, one of which can be used as a pollutant-absorbent material Islam et al., 2017. Hydrochar is a material that has a high carbon content and is rich in oxygenated functional groups such as hydroxyl, carbonyl, or carboxylate, with high hydrophilic properties and good chemical reactivity (Zhang et al., 2017; Mohadi et al., 2022b).

Hydrochar materials can be prepared from biomass, which comes from plants and animals. Example of biomass includes agricultural products, plantations, organic waste, wood, rice husks, and fruit peels. Biomass is a material that is rarely utilized, so the abundant availability of biomass requires maximum effort to have more value. According to experts, biomass has been reported to contain various organic compounds that can increase its ability to absorb dye contaminants (alphanes). Peel of duku fruit (*Lansium domesticum*) that has gone through a hydrothermal carbonization process to produce hydrochar to be used as adsorbent. The hydrochar produced from duku peel is expected to have a large surface area, good porosity, and high adsorption capacity. The material was then characterized using Fourier Transform-Infra Red (FT-IR) analysis to analyze the functional groups contained in the adsorbent, X-Ray Diffraction (XRD) analysis to see the structural stability of the material, Brunauer Emmet Teller (BET) analysis to analyze the surface area, pore diameter and pore volume of the material and Scanning Electron Microscopy (SEM) analysis to analyze the surface topography and elements contained in the analyzed material.

2. EXPERIMENTAL SECTION

2.1 Preparation of Duku (*Lansium domesticum*) Fruit Peel (Islam et al., 2017)

Peel of duku fruit (*Lansium domesticum*) was first sliced into small pieces and thoroughly washed to ensure cleanliness. Subsequently, the peel was sun-dried and further dried in an oven at 100°C for 8 hours. Afterward, the dried peel was crushed and passed through a 40-mesh filter to obtain the desired consistency.

2.2 Hydrothermal Carbonization (Hasanah et al., 2023; Wijaya et al., 2021)

A total of 2.5 grams of duku fruit (*Lansium domesticum*) peel, along with 50 mL of water, were introduced into a 100 mL Hydrothermal Stainless-steal Autoclave. Subsequently, the mixture was subjected to heating at temperatures of 200°C and 250°C, with varying durations of 8, 10, 12, and 24 hours. After the hydrothermal process, the resulting hydrochar was allowed to cool at room temperature for 12 hours and then washed with distilled water. To finalize the production of hydrochar products, they were dried in an oven at 105°C for 24 hours. The obtained hydrochar products were then subjected to several characterization techniques, including X-Ray Diffraction (XRD) analysis, Fourier Transform Infra Red (FT-IR) spectroscopy, Brunauer Emmet Teller (BET) analysis, and Scanning Electron Microscope (SEM) analysis.

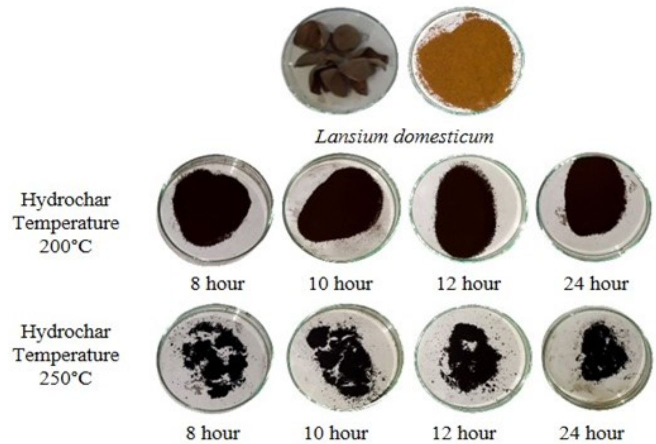

Figure 1. The Physical Form of Hydrochar Resulting from Carbonization by Variation of Carbonization Time and Temperature at Temperatures of 200°C and 250°C

2.3 Characterization Data Analysis

Peel of duku fruit (*Lansium domesticum*) and hydrochar are characterized using FT-IR, XRD, SEM, and BET analysis. This analysis is carried out so that it can be used as an indicator of the success of hydrochar material synthesis. Fourier Transform Infra-Red (FT-IR) analysis is used to determine the functional groups contained in the peel of duku fruit (Lansium domesticum) and hydrochar. X-Ray Diffraction (XRD) analysis was used to determine the level of crystallinity in the material on duku fruit peel (*Lansium domesticum*) and hydrochar. Scanning Electron Microscope (SEM) analysis is used to determine the morphological structure of the material in duku fruit peel (Lansium domesticum) and hydrochar. While Brunauer Emmet Teller (BET) analysis will be used to determine the surface area of the material on the peel of duku fruit (*Lansium domesticum*) and hydrochar (Sartika et al., 2014; Palapa et al., 2023a).

3. RESULTS AND DISCUSSION

3.1 Hydrothermal Carbonization of Duku Fruit Peel (*Lansium domesticum*)

The hydrothermal carbonization process aims to convert biomass into hydrochar which has a high carbon content. Figure 1 shows the biomass of duku fruit peel (*Lansium domesticum*) and hydrochar after carbonization, at various temperatures and times the hydrothermal carbonization process resulted in a change in the color of the biomass from brownish yellow to black. This is because the biomass has been converted into carbon material so that it changes color to black.

The hydrothermal carbonization process was carried out at temperatures of 200°C and 250°C with time variations of 8, 10, 12, and 24 hours. The temperature and time variations were carried out to obtain the quantity of % yield of the resulting product and the success of the hydrothermal carbonization process. Figure 2 presents the % yield from the hydrothermal carbonization process of biomass from duku fruit peel. Based on the graph presented in Figure 1, it shows that at 200°C, the % yield values resulting from the hydrothermal carbonization process are 60.66%, 58.82%, 55.07%, and 53.30% respectively, while at 250°C the % yield values produced are 42.53%, 38.73%, 36.41% and 35.83% respectively. The data in Figure 2 shows that different temperatures can affect the quantity of product produced. The higher the temperature and the longer the carbonization time used in the hydrothermal carbonization process causes the % yield produced by the product to decrease. This is because the higher the temperature and the longer the carbonization time causes the water content in the biomass to shrink or what is commonly called dehydration. In addition, high temperatures will cause the decomposition of volatile organic compounds other than carbon contained in biomass such as flavonoids, terpenoids, isoflavones, and lignin (Wijaya et al., 2023; Palapa et al., 2023b).

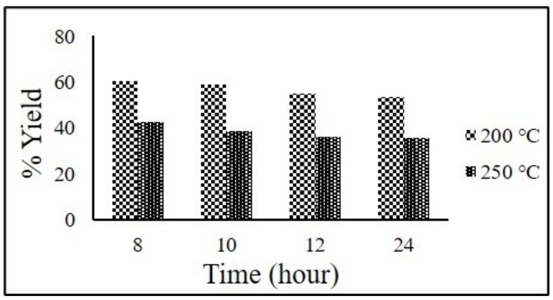

Figure 2. % Dry Weight Yield of Hydrothermal Carbonization Resulted Hydrochar Material

3.2 Fourier Transform Infra Red (FTIR) Analysis of Duku Fruit Peel (*Lansium domesticum*) and Hydrochar

Fourier Transform Infra-Red (FT-IR) analysis aims to detect any changes in functional groups contained in duku fruit peel biomass, and hydrochar. Figures 3a and 3b present the FT-IR spectra of duku fruit peel biomass and hydrochar at 200°C and 250°C with a variation of carbonization time for 8, 10, 12, and 24 hours. Figures 3a and 3b present the FT-IR spectra of various hydrothermal carbonization time treatments of duku fruit peel biomass with a magnification of the wave number scale of 500-4000 cm^{-1}. Based on the FT-IR spectra shown in Figures 3a and 3b, there is a peak with a strong intensity that appears at a wave number of about 3425.58 cm^{-1} in various treatments of duku fruit peel with temperature variations of 200°C and 250°C which indicates the presence of -OH groups from alcohols and carboxylic acids in the material (Mohadi et al., 2022a).

The peak tends to weaken as the carbonization time and temperature increase. The vibrational peak that appears at wave number 2931.80 cm^{-1} indicates the presence of aliphatic -CH groups from the vibrations of alkane compounds. The peak continues to increase in intensity as the carbonization time increases. In hydrochar with a carbonization temperature of 250° C, a sharper intensity appears in the treatment for 8 hours. When the treatment time is added there is a decrease in the intensity of the resulting vibration peak. A small peak also appears at a wave

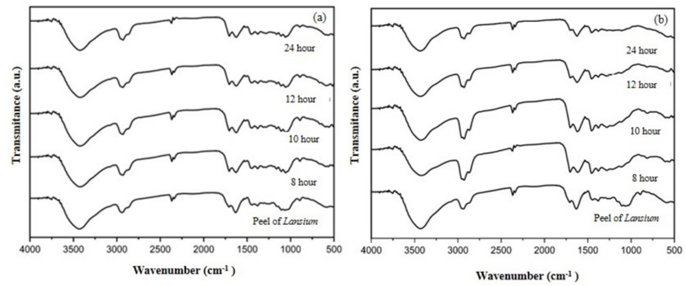

Figure 3. FT-IR Spectrum of Biomass and Hydrochar at (a) 200°C, and (b) 250°C

number of 2870.08 cm^{-1} indicating the presence of aliphatic =CH groups that appear due to the vibration of the alkene chain which is getting sharper as the carbonization time of duku fruit peel increases. When the carbonization temperature treatment was 250°C, the peak weakened at carbonization times of 12 and 24 hours. The peak that appears at wave number 2337.72 cm^{-1} indicates the presence of aromatic -CH groups derived from benzene in the material. Vibration peaks tend to increase with increasing treatment time at 200°C. However, in Figure 3b at a temperature of 250° C, the vibration peaks that appear tend to decrease in intensity, which can indicate a change in the arrangement of the carbon structure in the material. Vibration peaks with weak intensity at wave number 2137.17 cm^{-1} indicate the presence of bonds between carbon compounds derived from alkyne group compounds.

Figures 3a and 3b present the FT-IR spectra of various hydrothermal carbonization treatments of duku fruit peel. The sharp peak that appears at wave number 1635.64 cm^{-1} with two specific peaks indicates the presence of C=O groups derived from the vibrations of primary amide group compounds which tend to weaken with increasing carbonization time and temperature. The peak that tends to be stable at wave number 1442.75 cm^{-1} indicates the presence of a C=C stretching group derived from the vibration of aromatic compounds. The weak peak at wave number 1064.71 cm^{-1} indicates the presence of Si-O-Si functional groups whose intensity tends to disappear along with the longer treatment and the higher carbonization temperature. Vibration peaks with intensities appearing at wave numbers 894.97 cm^{-1}, 578.64 cm^{-1}, 516.92 cm^{-1}, and 470.63 cm^{-1} come from vibrations of carbon compounds bound to halide group compounds such as C-Cl, C-Br, C-I which tend to be maximal in hydrothermal treatment for 12 hours at 200°C and disappear in the treatment of longer time and higher temperatures. In general, based on the FT-IR spectra produced, it can be seen that several compounds are decomposed along with the increase in carbonization time and temperature. Table 1 shows the functional groups of the decomposed compounds that appear with increasing carbonization treatment.

Table 1. Compound Vibrations in FT-IR Spectra at Various Carbonization Time and Temperature Treatments

Function Group	Compound	Wave Numbers (cm^{-1})	Descriptions
-OH	Alcohol	3425.58	Peaks that tend to weaken as carbonization time increases
-CH	Alkanes	2931.80	Peaks that continue to increase as the carbonization time increases
=CH	Alkanes	2870.08	Small peaks that continue to increase as the carbonization time increases
-CH	Aromatics	2337.72	The resulting peak tends to increase as the carbonization time increases.
C=O	Amida	1635.64	Tends to weaken as the carbonization time treatment increases
C=C	Aromatics	1442.75	Stable peaks in each carbonization time treatment at 250°C.
Si–O–Si	Silicates	1064.71	Peaks that tend to disappear as carbonization time and temperature increase.
C–Cl		894.97	
C–Br	Halida	578.64	Peaks that tend to be maximum in the treatment of carbonization time of 12 hours with a temperature of 200 ° C.
C–I		470.63	

3.3 X-Ray Diffraction (XRD) Analysis of Duku Fruit Peel (*Lansium domesticum*) Material and Hydrochar

X-Ray Diffraction (XRD) analysis is employed to identify alterations in the crystal structure of biomass and hydrochar resulting from the hydrothermal carbonization procedure. Biomass that has gone through the hydrothermal carbonization process is expected to have a crystalline structure. Figures 4a and 4b present the XRD diffractograms of the duku (*Lansium domesticum*) fruit peel biomass and the repaired hydrochar with varying carbonization times of 8, 10, 12, and 24 hours at 200°C and 250°C, respectively. Based on the diffractograms in Figures 4a and 4b, two specific peaks are appearing at diffraction angles of 16.9° and 21.8° for the duku (*Lansium domesticum*) peel material (Zbair et al., 2020).

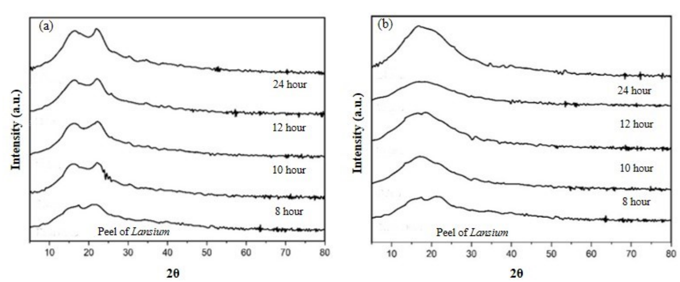

Figure 4. XRD diffractograms of Duku Fruit Peel and Hydrochar at Temperatures (a) 200°C and (b) 250°C

In Figure 4, the diffraction angles that appear in the hydrothermal carbonization process with carbonization time treatment for 8, 10, 12, and 24 hours are 15.7° and 22.59° for 8 hours treatment, 15.5°, and 22.50° for 10 hours treatment, 16.0°, and 22.47° for 12 hours treatment and 21.61° and 34.9° for 24 hours treatment, respectively. The peaks produced at these angles are specific to cellulose compounds. The variation of carbonization time given during the hydrothermal carbonization process generally did not change the diffraction peaks produced. This indicates that the hydrothermal carbonization process at 200°C does not change the structure of cellulose compounds. A slightly different thing was found in the carbonization process at 250°C where the resulting diffraction peaks experienced a slight change and only one diffraction peak was produced as presented in Figure 4b.

Figure 4b shows the diffractogram pattern produced at 250°C with a variation of carbonization time for 8, 10, 12, and 24 hours

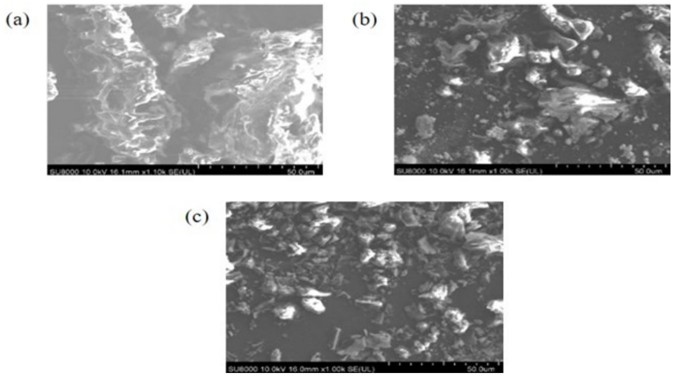

Figure 5. SEM Analysis Result of Material (a) Duku Fruit Peel (Lansium domesticum), (b) Hydrochar 200°C and (c) Hydrochar 250°C

resulting in diffraction angles of 30.9° for 8 hours treatment, 18.92° for 10 hours treatment, 19.166° for 12 hours treatment and 16.2° and 20.3° for 24 hours treatment, respectively. The carbonization process at higher temperatures causes a decrease in the level of crystallinity of the hydrochar material as indicated by the widening of the peak produced at 250°C. This resulted in the hydrochar material produced at 250°C tending to form a higher amorphous phase than at 200°C. The shift in angle and decrease in diffraction intensity indicate that during the hydrothermal carbonization treatment, the chemical compounds in the duku peel began to degrade but have not yet changed their crystal structure.

Based on Figures 4a and 4b, the longer the treatment is given, the narrower the resulting peak. This indicates that the expected material is increasingly forming a crystalline structure but is still in an amorphous state. Based on the peaks produced from both diffractograms, the best material at 200°C was produced when treated for 12 hours, while at 250°C the best material was produced when treated for 10 hours. This can also be seen from the small peak that appears in the diffraction angle region around 26° at 200°C, while in the carbonization treatment for 24 hours, the small peak in the diffractogram in the 26° region begins to disappear. At a temperature of 250°C, the best material was produced in the carbonization treatment for 10 hours where the resulting diffractogram pattern still resembles the diffractogram pattern of duku fruit peel (*Lansium domesticum*) material without undergoing the hydrothermal carbonization process, while in the 12 and 24 hour treatment, the resulting hydrochar material only forms one specific peak (Siregar et al., 2022).

3.4 Scanning Electron Microscopy (SEM) Analysis of Duku Fruit Peel (Lansium domesticum) and Hydrochar Materials

The surface morphology, particle size, and crystallographic structure of the observed material were examined using Scanning Electron Microscopy (SEM) analysis. Figure 5 displays the outcomes of the SEM analysis for the duku fruit peel biomass adsorbent and hydrochar treated at temperatures of 200°C and 250°C.

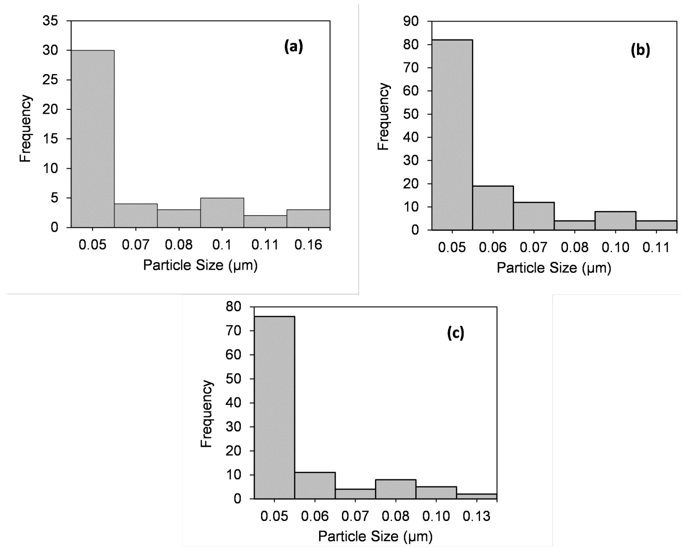

Figure 6. Particle Distribution Diagram of Material (a) Duku Fruit Peel, (b) Hydrochar at 200°C and (c) at 250°C

Based on Figure 5, the analysis results using SEM analysis show that the duku fruit peel (*Lansium domesticum*) material has different morphologies from each carbonization treatment and activation at 10.0 kV magnification. In Figure 5(a), the morphological pattern of the duku peel adsorbent particles demonstrates a tendency to aggregate or clump together in the same phase. On the other hand, Figures 5(b) and 5(c) exhibit the morphological pattern of particles from hydrochar adsorbents at 200°C and 250°C, respectively. These hydrochar particles appear to be heterogeneous and possess irregular shapes, likely as a result of the hydrothermal carbonization treatment applied, leading to particle breakage or deaggregation (Tondl et al., 2020).

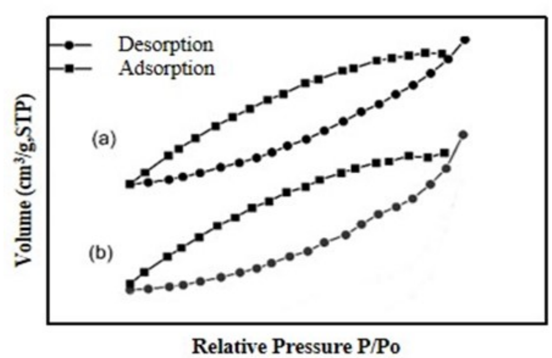

Figure 7. N_2 Adsorption Desorption Isotherm Graph (a) Duku Fruit Peel Biomass, (b) hydrochar at 200°C

Based on the diagram presented in Figure 6(a), it can be concluded that the duku fruit peel (*Lansium domesticum*) material has an average particle size distribution of 0.05 micrometers with a total of 30 particles distributed, while at a size of 0.07

Table 2. Measurement Data of BET Adsorption Desorption Isotherm of Biomass Material and Hydrochar at 200°C

Materials	Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Diameter (nm)
Biomass	12.343	0.022	2.647
Hydrochar (200°C)	22.635	0.044	2.769

micrometers, there are 4 particles, at a size of 0.08 there are 3 particles, at a size of 0.10 there are 5 particles, at a size of 0.11 there are 2 particles and at a size of 0.16 there are 3 particles. The uneven distribution is because the particles in the material tend to aggregate or accumulate with each other to form an aggregate as a result of the intramolecular interaction of the particles. Figures 6(b) and 6(c) present the particle distribution diagram of the hydrochar material at 200°C and 250°C. Based on Figure 6(b) the hydrochar material at 200°C has an average particle size of 0.05 micrometers of 82 particles, while Figure 6(c) shows that the hydrochar material at 250°C has an average particle size of 0.05 micrometers of 76 particles. The uneven particle distribution is due to the hydrothermal carbonization treatment given causing the particles in the material to tend to be heterogeneously irregular or deaggregated.

3.5 Brunauer Emmet Teller (BET) Analysis of Duku Fruit Peel Material (Lansium domesticum) and Hydrochar

Characterization analysis using the Brunnaeur Emmet Teller (BET) method aims to determine the surface area, pore volume, and pore diameter of the material used. In the analysis using the Brunnaeur Emmet Teller (BET) method, an adsorption-desorption isotherm graph was obtained for each material analyzed Figure 7 presents the adsorption-desorption isotherm graph of the duku fruit peel biomass material and hydrochar at a carbonization temperature of 200°C.

Based on the graph presented in Figure 7 shows the adsorption-desorption isotherm pattern that does not overlap each other from each material or what is commonly called hysteresis, this shows that the biomass material of duku fruit peel (*Lansium domesticum*), hydrochar at 200°C carbonization temperature is classified into type IV adsorption-desorption isotherm (Ulfa, 2016). Materials exhibiting mesoporous structures possess a distinctive feature known as Type IV adsorption isotherm, which is evident through the appearance of hysteresis between the adsorption and desorption curves. This hysteresis is a result of the presence of unevenly sized pores, leading to different directions in the resulting curves. The data obtained from BET measurements, including surface area, pore volume, and pore diameter for the three adsorbents, are presented in Table 2.

Based on the data presented in Table 2, the biomass of duku fruit peel (*Lansium domesticum*) has a surface area of 12.343 m^2/g with a pore volume reaching 0.022 cm^3/g and a pore diameter of 2.647 nm. The hydrothermal carbonization process carried out at 200°C increased the surface area of the material to 22.635 m^2/g which was accompanied by an increase in pore volume and pore diameter of the material (Congsomjit and Areeprasert, 2021).

4. CONCLUSIONS

The process of preparing duku fruit peel (*Lansium domesticum*) biomass using the hydrothermal carbonization method has been effectively accomplished. FT-IR analysis results showed the presence of vibrations that continue to increase at certain wave numbers indicating the presence of -OH, -CH, =CH, C=C, Si-O-Si, and Aromatic groups. XRD analysis showed an increasing diffraction peak at a diffraction angle between 20.50° and decreasing at a diffraction angle of 15.5° with activation treatment. BET analysis showed that the hydrochar at 200°C carbonization temperature had a larger surface area than the biomass but the higher carbonization temperature caused structural damage to the material. SEM analysis shows the surface morphology that is increasingly hollow from each carbonization treatment.

5. ACKNOWLEDGEMENT

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