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Characterization of activated micro biochar form rice husk pyrolysis with hydrochloric acid solution activation

Shafa Salsabila Aji[*1], Nimas Mayang Sabrina Sunyoto[1], Hendrix Yulis Setyawan[1]
[1] *Department of Agroindustrial technology, Faculty of Agricultural Technology, Brawijaya University*
*Corresponding author: shafasalsabila@student.ub.ac.id

Abstract

Rice husk is the result of rice grain milling which is obtained around 20-30% of the initial weight. This high percentage of husk waste has the potential to be processed into activated biochar because it is rich in organic materials, especially carbon. Activated biochar is a porous material with a large surface area, so it can be utilized for various applications. This study aims to analyze the influence between 2 factors, namely variations in HCl activator concentration (1M, 1.5M, 2M) and soaking time (12 hours, 18 hours, 24 hours) on rice husk biochar. After that, characterization was carried out which included testing water content, ash content, volatile matter content, fixed carbon content, iodine number, yield, and BET surface area. The best treatment was also subjected to FTIR test and determined using the zeleny method. The results showed that rice husk biochar has a moisture content between 1.12%-2.24%, ash content between 48.45%-50.01%, volatile substance content between 11.65%-16.23%, fixed carbon content between 34.20%-40.66%, iodine number between 325.18mg/g-470.98mg/g, yield between 81.34%-87.42%, and BET surface area between 358.61m^2/g-519.40m^2/g. Biochar with the best characteristics was obtained in the treatment of 2M HCl Concentration and 24 hours of soaking time.The treatment has characteristics of moisture content of 1.20%, ash content of 48.62%, volatile matter content of 11.65%, bound carbon content of 40.66%, iodine number of 470.98mg/g. This study highlights the physico-chemical properties of biochar with potential for further applications in agricultural technology, waste treatment, and as an eco-friendly material.

Keywords: *Rice Husk, Micro Biochar, Chemical Activation*

Introduction

Rice production in Indonesia has increased from 54,415,294.22 tons in 2021 to 54,748,977 tons in 2022 (BPS, 2022). This increases the capacity of rice husk waste, which has a composition of 20-30% of the results of rice milling (Patabang, 2012). This high percentage of husk waste is usually just thrown away and becomes an environmental problem due to limited disposal sites, processing technology and science.

According to research by Soltani et al. (2015), rice husk is rich in carbon by 37.05%, hydrogen by 8.80%, nitrogen by 11.6%, and oxygen by 35.03%. Rice husk also contains lignocellulose, namely cellulose about 36-40g/100g, hemicellulose about 12-19g/100g, lignin about 20g/100g, and ash about 12g/100g (Nasciemto et al., 2015). High carbon content can be utilized as biochar which can be applied as an adsorbent for heavy metals and organic compounds of industrial waste (Syauqiyah et al., 2011),

phenol removal (Yulianti and Susanto, 2011) and even as an adsorbent for CO and NO gas emissions from motor vehicles (Febriyanti et al., 2020). The use of biochar as fertilizer can improve the chemical, biological and physical properties of soil. In addition, the addition of biochar to the soil increases the C-Organic content, as well as provides N, P, and K nutrients and remediates the soil (Putri et al., 2017). The application of biochar becomes less optimal because of the small surface area, so it is necessary to expand the surface by making micro biochar. Micro biochar is biochar with micro size (<100µm) (Lonappan et al., 2020).

The process of making activated micro biochar consists of two stages, namely the pyrolysis process and the activation process. Pyrolysis is a process of converting complex hydrocarbon compounds into small molecules. There are three products resulting from this process, namely solids (biochar), liquids (bio-oil), and gas (syngas) (Goenadi and Santi, 2017). After pyrolysis, biochar is activated to have a larger surface, so that its adsorption power is greater. This is because soaking with chemical solutions can cause impurities on the surface of biochar to decompose (Masthura and Zulkarnain, 2018). Research that discusses activated micro biochar using HCl activator is still limited. In addition, the use of activation with HCl on rice husk biochar has also not been done. Therefore, it is necessary to conduct research that examines the effect of activator concentration and soaking time with HCl solution on the characteristics of activated micro biochar rice husk products.

Research Method

Tools and Materials

Tools used in this research include pyrolysis machine, analytical balance, ball mill, blender, 300 mesh sieve, furnace, hot plate stirrer, oven, desiccator, crucible cup, porcelain cup, tongs, beaker glass, erlenmeyer, measuring cup, funnel, and stirrer. Materials used in this research include rice husk, distilled water, HCl solution, 0.1N iodine solution, filter paper, plastic clips, aluminum foil, and label paper.

Research Design

The research design used is a Randomized Block Design using two factors, each factor consisting of 3 levels. HCl concentration factor (A Factor), consisting of 1M, 1.5M, and 2M treatments. Soaking time factor (B Factor), consisting of soaking time for 12, 18, and 24 hours. In this study, a negative control was used which was a biochar sample without an activation process or micro biochar that was not activated. This control sample is used as a comparison to determine the difference in factors or research treatments. The Randomized Group Design can be seen in Table 1.

Tabel 1. Randomized Block Design

A Factor	B Factor	Reply		
		1	2	3
A1	B1	$A1B1_1$	$A1B1_2$	$A1B1_3$
	B2	$A1B2_1$	$A1B2_2$	$A1B3_3$
	B3	$A1B3_1$	$A1B3_2$	$A1B3_3$
A2	B1	$A2B1_1$	$A2B1_2$	$A2B1_3$
	B2	$A2B2_1$	$A2B2_2$	$A2B2_3$
	B3	$A2B3_1$	$A2B3_2$	$A2B3_3$
A3	B1	$A3B1_1$	$A3B1_2$	$A3B1_3$

A Factor	B Factor	Reply		
		1	2	3
	B2	$A3B2_1$	$A3B2_2$	$A3B2_3$
	B3	$A3B3_1$	$A3B3_2$	$A3B3_3$

Micro Biochar Preparation

Dry rice husk waste was weighed 500 g and pyrolyzed using a pyrolysis machine. The pyrolysis temperature was set to reach 600°C. Then the pyrolysis process continued for 2 hours (Modification of Prayogo method, 2023 and Wardalia et al., 2021). After the pyrolysis process, the biochar is reduced in size using a blender and ball mill at 100 rpm for 2 hours. Then, sieved with a 300 mesh sieve to produce micro size. Residues that do not pass the sieve are ball milled again and sieved again (Modification of Pamungkas method, 2023).

Micro Biochar Activation Process

The micro biochar powder obtained from the previous process was then weighed 20 g and soaked in HCl activator solution (1M; 1.5M; and 2M) with a biochar:distilled water ratio of 1:10 (w/v) for 12, 18, and 24 hours at room temperature. The soaked biochar was then separated from the activating solution using filter paper and washed with distilled water until a neutral pH was obtained. Activated micro biochar was then dried in an oven (Memmert) at 100°C for 3 hours and analyzed for moisture content, ash content, volatile substance content, bound carbon content and iodine absorbency (Modified method Prayogo, 2023 and Verayana et al., 2018).

Test Procedure

a. Water Content

Testing the water content aims to determine the amount of water contained in the material. The smaller the value of water content in the material, the better the quality of activated micro biochar. The water content test uses the gravimetric principle and refers to SNI 06-3730-1995 and Dewantisaraswati (2019). Moisture content is calculated using the weight of 1 g sample. The sample was oven dried at 105°C for 3 hours. Then, the sample was cooled in a desiccator until the temperature stabilized ± 15 minutes and weighed using an analytical balance until constant weight. Moisture content was calculated using equation 1.

$$\text{Moisture Content (\%w/w)} = \frac{W1-W0}{W1-W2} \text{ x } 100\% \text{.....(1)}$$

Description:
W0 = empty cup weight
W1 = sample weight + cup before heating (g)
W2 = sample weight + cup after heating (g)

b. Ash Content

Ash content testing aims to determine the amount of ash contained in the sample after ignition. The smaller the ash content value, the better the quality of activated micro biochar. The moisture content test uses the gravimetric principle and refers to SNI 06-3730-1995 and Dewantisaraswati (2019). Ash content was calculated using the weight

of 1 g sample. The sample was dried in a furnace at 800°C for 2 hours. Then, the sample was cooled in a desiccator until the temperature stabilized ± 15 minutes and weighed using an analytical balance until constant weight. Ash content was calculated using **equation 2.**

$$\text{Ash Content (\%w/w)} = \frac{W1}{W2} \text{ x } 100\% \text{ (2)}$$

Description:
W1 = sample weight after combustion (g)
W2 = sample weight before combustion (g)

c. Volatile Matter

Volatile matter testing aims to determine the content of volatile substances when the heating temperature reaches 950°C. The smaller the value of volatile matter content, the better the quality of activated micro biochar. The water content test refers to SNI 06-3730-1995 and Dewantisaraswati (2019). Ash content is calculated using the weight of 1 g of sample in a porcelain cup that has known empty weight. Then, the cup was tightly closed and heated in a furnace at 950°C for 10 minutes. Then, the sample was cooled in a desiccator to a stable temperature and weighed with an analytical balance to a constant weight. Volatile matter content was calculated using equation 3.

$$\text{Volatile Matter Content } \left(\%\frac{w}{w}\right) = \frac{W1 - W2}{W2} \text{ x } 100\% \text{ ..... (3)}$$

Description:
W1 = initial sample weight before combustion (g)
W2 = sample weight after combustion (g)

d. Fixed Carbon Content

Fixed carbon content test aims to determine the amount of pure carbon that is still bound to the activated micro biochar. The greater the value of fixed carbon content, the better the quality of activated micro biochar. The fixed carbon content test refers to SNI 06-3730-1995 and Dewantisaraswati (2019). Fixed carbon content is calculated using equation 4.

Fixed carbon content (% w/w) = 100% – %ash content – %volatile matter content.....(4)

e. Iodine Number

The iodine number test aims to determine the adsorption ability of activated micro biochar to iodine solution. The higher the absorbency of activated micro biochar to iodine solution, the better the quality. The test of iodine number using the titration principle refers to SNI 06-3730-1995. The steps are that the sample is first heated in an oven at 105°C for 1 hour. Then, 0.25 g of sample was put in an erlenmeyer and 25 ml of 0.1 N iodine solution was added, then stirred at room temperature for 15 minutes. Next, the sample was filtered with filter paper, so that the filtrate was obtained. The filtrate was taken as much as 10 ml using a measuring pipette and titrated with 0.1 N thiosulfate solution until light yellow in color. After that, a few drops of 1% amylum solution were

added and titrated again until the blue color in the solution disappeared. The iodine number test was calculated using equation 5.

$$\text{Iodine Number (mg/g)} = \frac{A-\left(\frac{B\ x\ N\ Na2S2O2}{N\ iodin}\right)x\ 12.6393\ x\ fp}{a}\ x\ 100\%.....(5)$$

Description:
A = Titration volume (mL)
B = Volume of Na2S2O2 used (mL)
fp = Dilution factor
a = Weight of activated charcoal
12.693 = amount of iodine corresponding to 1 ml of Na2S2O2 solution

f. The yield

The test aims to calculate the amount of activated carbon after the carbonation process (Egboiuba et al., 2019). In this study, a sample of 20 g was used which was then activated. After that, it was dried and weighed again to calculate the yield with equation 6.

$$\text{Yields} = \frac{Weight\ after\ activation}{Weight\ before\ activation}\ x\ 100\%.....(6)$$

g. Brunauer, Emmet, and Teller (BET) Surface Area

According to research by Hendrawan et al. (2021), in addition to using BET special testing, identification of activated carbon surface area can also be known using the iodine absorption approach. BET testing serves to measure the surface area and pore size distribution of activated carbon. BET surface area can be calculated using equation 7.

$$\text{BET } (m^2/g) = \frac{Iodine\ number}{538,9}\ x\ 594,3.....(7)$$

Data Analysis

Data obtained from the test results including yield, moisture content, ash content, volatile matter content, fixed carbon content, iodine number, surface area (BET), and yield were analyzed to evaluate the effect of each treatment on each characteristic. The data analysis method used was two-way ANOVA with a significance level of 5%. The software used for two-way ANOVA is SPSS 17. The results of ANOVA on each test characteristic will be evaluated to see whether there is a real effect of treatment or not. If there is an effect, then the test is continued with the Duncan's Multiple Range Test (DMRT) method to determine which treatment gives a different effect. Determination of the best treatment using the Multiple Attribute Zeleny method, using 5 parameters including iodine absorption of moisture content, ash content, volatile matter content, fixed carbon content, and iodine number. The ideal criteria for each parameter is based on the minimum and maximum limits stated in SNI 06-3739 1995.

Results and Discussion

Characteristics of Rice Husk Waste Micro Biochar

The results of biochar obtained from the pyrolysis process have physical characteristics, namely shiny black color, powder, dry, and smells typical of charcoal. The results of biochar that have been sifted can be seen in Figure 1.

Figure 1. Rice husk waste carbon

Analytical results include yield, moisture content, ash content, volatile substance content, bound carbon content and iodine number. The test results are shown in Table 1.

Table 1. Proximate Characteristics of Micro Biochar

Parameters	Average Result	SNI Standards
Moisture Content (%)	2,94 ± 0,09	Max. 15%
Ash Content (%)	50,69 ± 0,55	Max. 10%
Volatile Matter Content (%)	17,77 ± 0,61	Max. 25%
Fixed Carbon Content (%)	31,50 ± 0,58	Min. 65%
Iodine Number (mg/g)	323,240 ± 1,43	Min. 750 mg/g

Based on these results, the parameters that meet SNI standards are moisture content and volatile substance content, while ash content, bound carbon content and iodine absorbency do not meet SNI. The ash content produced is very high because the rice husk contains very high silica, which during the pyrolysis process will not burn and turn into solid silica ash (Abdurrohmansyah et al., 2015).

Characteristics of Activated Micro Biochar

Moisture Content

The average test results of rice husk micro biochar moisture content ranged from 1.12% - 2.24% and have met SNI which is ≤ 15%. The lowest water content results were shown in activated micro biochar activated in 2M HCl treatment for 18 hours at 1.12% and the highest was shown in 1M HCl treatment for 12 hours at 2.24%. The results of the two-way ANOVA test showed that the activator concentration factor and soaking time had a significant effect on moisture content with a significance level of $0.000 < \alpha$ (0.05) and $0.001 < \alpha$ (0.05), respectively. While the interaction between the two factors had no significant effect with a significance level of $0.828 > \alpha$ (0.05). The DMRT test results of the concentration factor showed that the 1M HCl treatment had the highest mean and was significantly different. While the DMRT test for the length of

soaking treatment 12 hours different has the highest mean and significantly different. The graph of activated micro biochar moisture content can be seen in Figure 2.

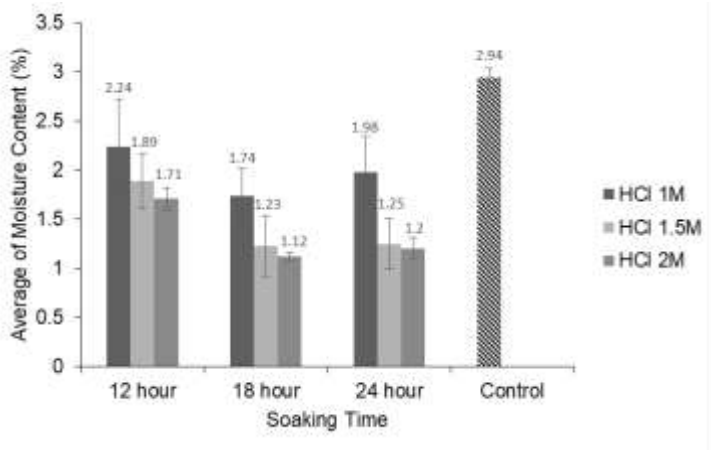

Figure 2. Graph of moisture content of activated micro biochar with standard error

Based on the figure, it can be seen that the control sample or sample without activation shows a higher water content than all samples after activation. This is because the activation process can reduce the water content. According to research by Esterlita and Herlina (2015) during activation, activator agents will bind water molecules to activated carbon, so that the water content can decrease.

The activator concentration factor shows a downward trend as the concentration increases. In theory, the higher the activator concentration, the lower the water content value. This is due to the influence of the hydroscopic nature of the HCl activator which can absorb the water content in the material (Verayana et al., 2018). According to Nurrahman et al. (2021), water content is assumed to be a volatile compound whose presence in large quantities will cover the pores of activated carbon. According to Nurfitria et al. (2019), volatile compounds include impurities that can be reduced using the activation process.

The soaking time factor shows a fluctuating trend. The decrease in water content is caused by the number of water molecules absorbed by the activator, in this case H+ ions. However, in the 24-hour immersion treatment, there was a slight increase in moisture content. According to research by Dwityaningsih et al. (2023), the increasing value of water content can also be caused because during neutralization, activated carbon has longer contact with distilled water, so that the water content becomes higher.

Ash Content

The average test results of the ash content of activated micro biochar of rice husk ranged from 48.45% - 50.01% and did not meet the SNI which is ≤ 10%. The lowest ash content was shown in activated micro biochar activated in 1.5M HCl treatment for 24 hours at 48.45% and the highest was shown in 1M HCl treatment for 12 hours at 50.01%. The results of the two-way ANOVA test showed that the activator concentration factor and soaking time had a significant effect on ash content with a significance level of $0.000 < \alpha$ (0.05) and $0.001 < \alpha$ (0.05), respectively. Meanwhile, the interaction between the two factors had no significant effect at $0.183 > \alpha$ (0.05). The DMRT test results of the concentration factor showed that the 1M HCl concentration

treatment had the highest average and was significantly different. While the DMRT test for the length of soaking showed that there were no significantly different results. The graph of activated micro biochar ash content can be seen in Figure 3.

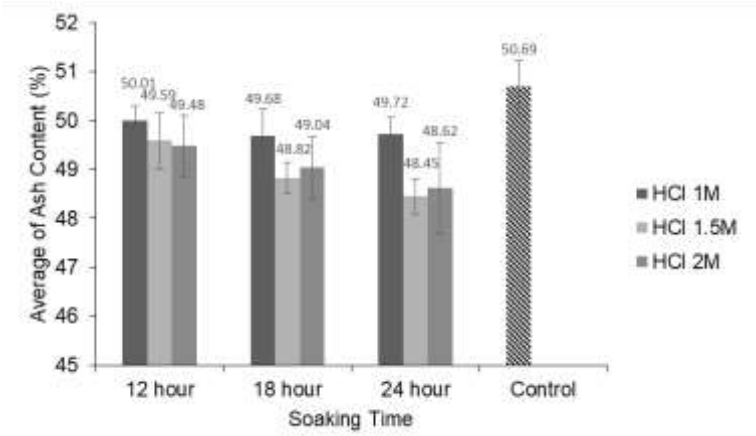

Figure 3. Graph of ash content of activated micro biochar with standard error

Based on this figure, it can be seen that the control sample or sample without activation shows a higher ash content than all samples after activation. This is because activated carbon before activation still contains a lot of organic minerals, so the ash content is high. According to Priambodo et al. (2016), activating agents have the ability to dissolve impurities such as ash, volatile content and tar contained in the pores of activated carbon.

The activator concentration factor shows a fluctuating data trend. The decrease in ash content is because the remaining minerals in activated carbon will mostly dissolve during the activation process, so that the carbon pores are also getting bigger (Batu et al., 2022). However, the results also show that the use of higher concentrations causes higher water content. This is because the higher the concentration of activator, the water content will tend to decrease. This results in mineral content that may form during the activation process becoming undissolved and still attached to the pores of activated carbon (Oko et al., 2021). In addition, it is caused by the lack of clean washing or neutralization process after activation (Rosalina et al., 2016).

The soaking time factor shows a data trend that tends to decrease as the soaking time increases. According to Kasmawarni (2013), the longer activation time causes the decomposition of complex compounds that close the pores of activated carbon, so that the ash left behind is purer and more porous activated carbon. Supported by research by Utomo (2016) which states that the longer the activation time, the inert substances on the surface of the carbon particles will be released from the surface.

Volatile Matter Content

The average test results of volatile matter content activated micro biochar rice husk ranged from 11.65% - 16.23% and has met SNI which is ≤ 25%. The lowest volatile matter content results were shown in activated micro biochar activated in 2M HCl treatment for 24 hours at 11.65% and the highest was shown in 1.5M HCl treatment for 12 hours at 16.23%. The results of the two-way ANOVA test showed that the activator concentration factor and the length of soaking had a significant effect on

the volatile substance content with a significance level of 0.018 < α (0.05) and 0.001 < α (0.05), respectively. While the interaction between the two factors showed no significant effect at 0.413 > α (0.05). The DMRT test results show that the activator concentration factor has no significant difference, while the soaking time factor shows the results of 24 hours soaking time has the lowest value and is significantly different. Graph of activated micro biochar volatile matter content can be seen in Figure 4.

Based on the figure, it can be seen that the control sample or sample without activation shows higher levels of volatile substances than all samples after activation. According to Zalmi and Khair (2021), the activation process can vaporize the levels of volatile matter. In addition, Alpian et al. (2020) also stated that the decrease in volatile matter levels is thought to be because the activated carbon no longer contains volatile smatter such as CO, CH4, H2, which evaporate during the activation process.

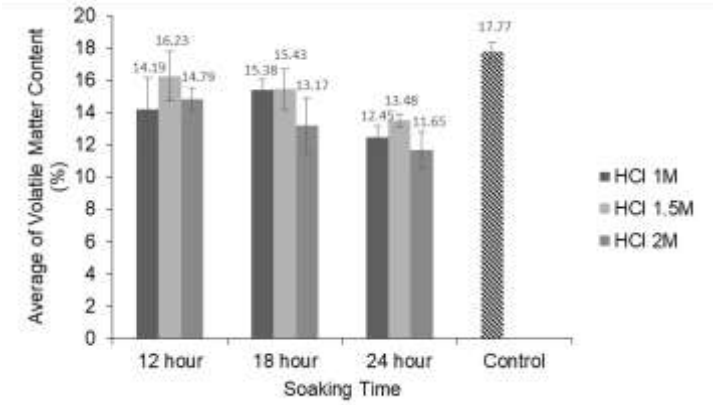

Figure 4. Graph of volatile matter content of activated micro biochar with standard error

The activator concentration factor shows a fluctuating data trend and tends to decrease as the activator concentration increases. This is because the addition of activator concentration can cause the pores of the carbon to be more open, so that the volatile substances contained therein will be pushed out (Hendrawan et al., 2017). Husin and Hasibuan (2020) also stated that the higher the concentration of activator, the residual hydrocarbon compounds attached to the pores of activated carbon come out. However, in the treatment of 1.5M HCl activator concentration, the volatile matter content increased. This is thought to be caused by the interaction between activated carbon and air, so that the levels of volatile matter increase.

The soaking time factor shows a data trend that tends to decrease as the soaking time increases. The length of soaking which tends to be longer, can result in carbon pores being eroded and wasted during the neutralization process, this is what makes the volatile matter content increase. according to the research of Wahyuni and Fathoni (2019), the value of volatile matter content will be directly proportional to the water content, in this study, the treatment of the two factors on water content showed a fluctuating data trend and tended to decrease, so that the value of volatile matter content also decreased.

Fxed Carbon Content

The average test results of fixed carbon content of activated micro biochar rice husk ranged from 34.20% - 40.66% and did not meet the SNI which is > 65%. The

lowest fixed carbon content was shown in activated micro biochar activated in 1.5M HCl treatment for 12 hours at 34.20% and the highest was shown in 2M HCl treatment for 24 hours at 40.66%. The results of the two-way ANOVA test showed that the activator concentration factor and soaking time had a significant effect on the fixed carbon content with a significance level of $0.004 < \alpha\ (0.05)$ and $0.000 < \alpha\ (0.05)$, respectively. While the interaction between the two factors showed no significant effect at $0.508 > \alpha\ (0.05)$. The results of the DMRT test for the concentration factor showed that the 2M HCl treatment had the highest average and was significantly different, while the soaking time factor showed that the 24-hour soaking time had the highest average and was significantly different. The graph of activated micro biochar fixed carbon content can be seen in Figure 5.

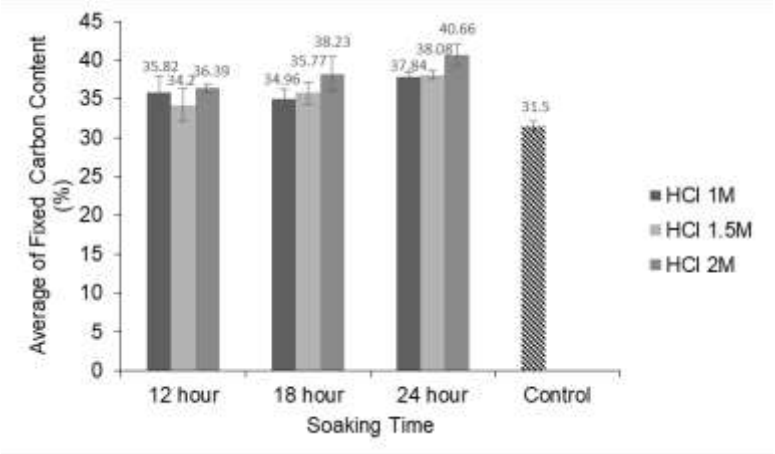

Figure 5. Graph of fixed carbon content of activated micro biochar with standard error

Based on the figure, it can be seen that the control sample or sample without activation shows lower levels of fixed carbon than all samples after activation. According to Sabandi et al. (2021), the fixed carbon content will increase after the activation process because the ash content and volatile matter content decrease. This is because the impurities that cover the carbon pores have disappeared due to the activation process, so that the pores become larger and the adsorption performance will be better than before activation (Dewi and Hidajati, 2012).

The activator concentration factor shows a fluctuating data trend, but tends to increase as the activator concentration increases. According to Simbolon et al. (2022), the increase in fixed carbon content is due to the carbonization process running perfectly, then the activated carbon will evaporate a lot of extractive substances until the level of volatile substances left behind is small and the fixed carbon content increases. However, at 1.5M HCl concentration there is a decrease in the value of fixed carbon content. This can be caused by the high ash content and volatile matter content obtained in the 1.5M HCl concentration treatment. which is inversely proportional and will reduce the fixed carbon content (Lestari et al., 2016).

The soaking time factor shows an upward data trend as the soaking time increases. The high value fixed carbon in activated carbon indicates a higher level of purity caused by the loss of non-carbon compounds during activation (Hendra et al., 2014).

Iodine Number

The average results of the iodine number test of activated micro biochar from rice husk ranged from 325.18 mg/g – 470.98 mg/g and did not meet SNI, which was > 750 mg/g. The lowest iodine number results were shown in activated micro biochar activated in 1.5M HCl treatment for 18 hours at 325.18 mg/g and the highest was shown in 2M HCl treatment for 24 hours at 470.98 mg/g. The results of the two-way ANOVA test showed that the concentration of the activator, soaking time and interaction between factors had a significant effect on iodine number with a significance level of 0.006 <α (0.05), 0.028 <α (0.05), and 0.002 <α (0.05). The DMRT test results of the concentration factor showed that 2M HCl had the highest average and was significantly different, while the soaking time factor showed that the results of the 24-hour soaking time had the highest average and were significantly different. Meanwhile, the DMRT test for the interaction between the two factors was categorized into 3 different subsets which can be seen in Figure 6.

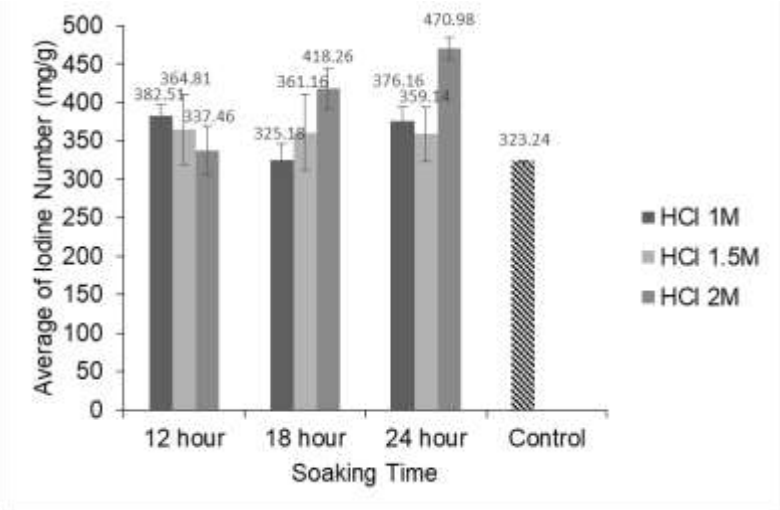

Figure 6. Graph of iodine number of activated micro biochar with standard error

Based on the image, it can be seen that the control sample or sample without activation showed a lower iodine absorption capacity compared to all samples after activation. According to Utomo (2014), this is because in the activation process, many inert substances on the carbon surface will be released, so that the pores will increase and the surface area will be larger, thus increasing the iodine absorption capacity.

The activator concentration factor shows a fluctuating data trend and tends to increase with increasing concentration. The high activator used can cause many minerals to be adsorbed and is able to remove more hydrocarbon deposits (Rizkyi et al., 2016). However, based on the graph, it can also be seen that there is a decrease in iodine absorption capacity when using 1.5M HCl and 2M HCl concentrations at a soaking time of 12 hours. This is due to the correlation between iodine absorption capacity and high fixed carbon content and ash content. According to Zalmi and Khair (2021), the lower the ash content and moisture content, the more carbon structures will be formed.

The soaking time factor shows a fluctuating data trend and tends to increase with the soaking time. According to Sa'diyah and Lusiani (2022), this increase can occur because the longer the activation, the longer the contact time between the activator and

carbon, allowing the binding of more impurities. This causes the pores of the activated carbon to open more and expand its surface, so that iodine absorption can run more optimally.

Yield

The yield analysis aims to determine the amount of activated micro biochar obtained from rice husk waste after the pyrolysis and activation processes. The average yield test results ranged from 81.34% - 87.42%. The lowest yield test results were shown in activated micro biochar activated in 2M HCl treatment for 18 hours at 81.34% and the highest was shown in 1M HCl treatment for 12 hours at 87.42%. The results of the two-way ANOVA test showed that the concentration factor of the soaking activator had a significant effect on the yield with a significance of 0.037 <α (0.05). Meanwhile, the soaking time factor and the interaction of the two factors did not have a significant effect with a significance level of 0.183 <α (0.05) and 0.255 <α (0.05), respectively. The results of the DMRT concentration factor test showed that the highest average yield was 2M HCl but was not significantly different from the others. The activated micro biochar yield graph can be seen in Figure 7.

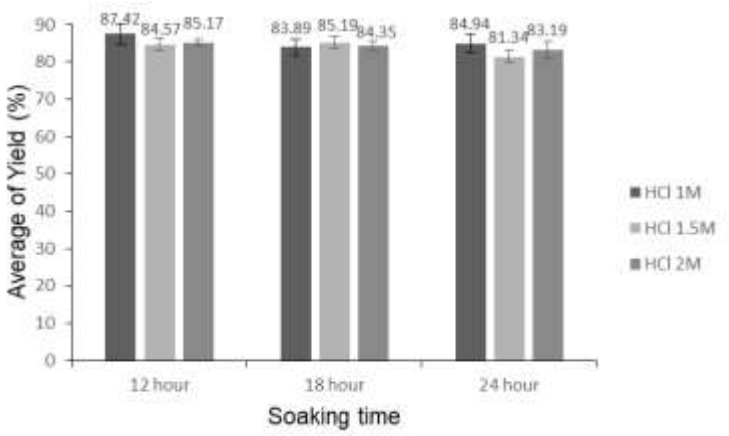

Figure 7. Graph of activated micro biochar yield with standard error

The concentration factor shows a fluctuating data trend and tends to decrease with increasing concentration. According to Erlina et al. (2015), the greater the concentration of the activator, the greater the degradation of the material that occurs, so that the yield value will decrease. Permatasari et al. (2014) also stated that the decrease in the percentage of activated carbon yield is because volatile substances and impurities such as mineral salts have been lost during the activation process, so that the resulting yield tends to be low.

Meanwhile, the soaking time factor does not have a significant effect on the yield as indicated by fluctuating data and tends to increase at a soaking time of 24 hours. This may be because the solution has reached its saturation point or the density of the solution is too large compared to the mass of the soaked charcoal, so that more charcoal will float on the surface (Erlina et al., 2015).

Surface Area (BET)

The BET test aims to measure the surface area and pore size distribution of activated carbon. The average surface area test results of rice husk activated micro

biochar ranged from 358.61 m2/g – 519.40 m2/g. The smallest surface area results were shown in activated micro biochar activated in 1M HCl treatment for 18 hours at 358.61 m2/g and the largest was shown in 2M HCl treatment for 24 hours at 519.40 m2/g. The results of the two-way ANOVA test showed that the activator concentration factor, soaking time and interaction between factors had a significant effect on iodine absorption with a significance level of 0.006 <α (0.05), 0.028 <α (0.05), and 0.002 <α (0.05). The DMRT test results of the concentration factor showed that 2M HCl had the highest average and was significantly different, while the soaking time factor showed that the results of the 24-hour soaking time had the highest average and were significantly different. Meanwhile, the DMRT test for the interaction between the two factors was categorized into 3 different subsets which can be seen in Figure 8.

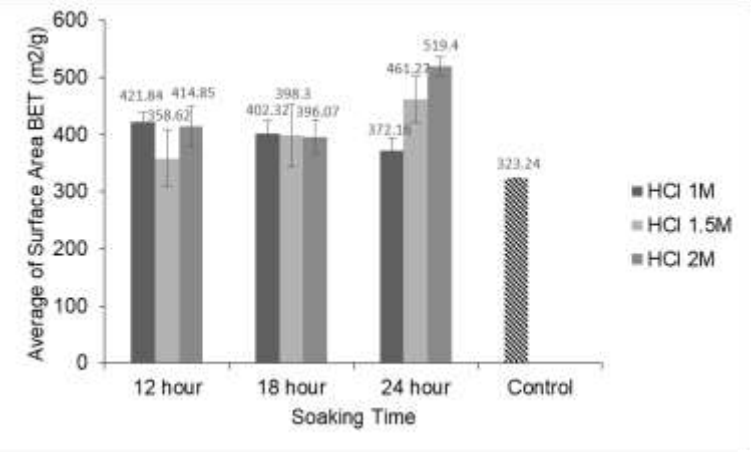

Figure 8**.** Graph of surface area of activated micro biochar with standard error

Based on the image, it shows a fluctuating data trend and tends to increase with increasing HCl concentration and soaking time. This is because the more activator is added, the more impurities are bound, so that it will open the activated carbon pores more. According to Nurdin et al. (2022), the activation process can create porosity and enlarge pores that are directly related to the surface. However, at some points there is a decrease in surface area such as in 1M HCl for 24 hours. It is suspected that the longer the soaking time or excessive activation time can break down micropores into meso- or even macropores (Yang et al., 2017).

Determination of the Best Treatment

Determination of the best treatment of activated micro biochar was carried out using the Multiple Attribute Zeleny method. The parameters used were water content, ash content, volatile matter content, bound carbon content, and iodine absorption capacity. Activated micro biochar from rice husk waste was not compared directly to determine the best treatment, but was seen from the ideal value obtained. The minimum ideal value was used for parameters such as water content, ash content, and volatile matter. While the maximum ideal value was used for the parameters of bound carbon content and iodine absorption capacity.

The best treatment results were obtained from the treatment of 2M HCl activator concentration with a soaking time of 24 hours. While the treatment that gave the worst

results was the 1M HCl concentration factor with a soaking time of 12 hours. A comparison of the best treatment with the established standards is shown in Table 2.

Table 2. Comparison of the best treatment results with SNI standards

Parameters	Mean Result	SNI Standards
Moisture Content (%)	1,20%	Max. 15%
Ash Content (%)	48,62%	Max. 10%
Volatile Matter Content (%)	11,65%	Max. 25%
Fixed Carbon Content (%)	40,66%	Min. 65%
Iodine Number (mg/g)	470,98%	Min. 750 mg/g

Based on the table, it can be seen that the characteristics of activated micro biochar with the best treatment that meets SNI parameters are water content and volatile matter content. Meanwhile, ash content, fixed carbon content and iodine number have not met SNI standards. As previously known, the high ash content in this study was largely influenced by the biomass used, namely rice husks, which contain high silica. During the combustion process, the silica content will not burn, but will turn into silica ash (Abdurrohmansyah et al., 2015). This high ash content affects the fixed carbon content and iodine number. The higher the ash content, the fixed carbon content and iodine number will decrease. This is because impurities such as mineral salts cover the pores of activated micro biochar, so that the surface area is smaller (Febrina and Rizki, 2023). The best treatment results obtained were tested using Particle Size Analyzer (PSA) and Fourier Transform Infrared (FTIR). The PSA test results are used to ensure that the particle size of the activated biochar produced is micro. Meanwhile, the FTIR test results were used to determine and identify the presence of functional groups from activated micro biochar (Pandia and Warman, 2016). The PSA test results obtained for the best treatment were 2.284 µm. Based on these results, it is proven that the sample has a micro size. According to research by Lonappan et al. (2020), biochar can be said to be micro if it has a size of <100µm. Meanwhile, the FTIR test results obtained can be seen in Figure 9.

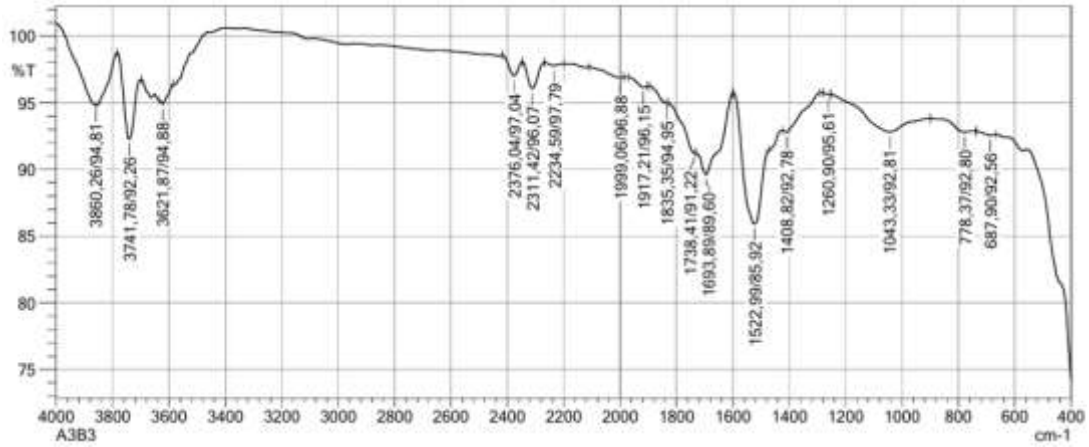

Figure 9. FTIR Test Results

Based on the image, it can be seen that there are peaks that indicate the presence of activated carbon functional groups. The functional groups found in the sample are

determined by looking at the peaks in the cm-1 range. According to Dahliyanti et al. (2019), the steeper the peak, the higher the functional group. The functional groups based on the FTIR peaks can be seen in Table 3.

Table 3. Functional Groups Based on FTIR Peaks

The Peak	Wavelength (cm^{-1})	Functional Groups and Wavelength (cm^{-1})	Reference
1	3860,26		
2	3741,78	O-H Hidroksil (3800-3300)	Zhang et al., 2020
3	3621,87		
4	2376,04		
5	2311,42	C≡C Karbon rangkap tiga (2340-2960	Harmita, 2012
6	2234,59		
7	1999,06		
8	1917,21	C≡O karbonil (1800-2300)	Tiwow et al., 2021
9	1835,35		
10	1738,41	C=O Karboksil (1700-1740)	Cabaniss et al., 1998
11	1693,89		
12	1522,99	N=O (1500-1600)	Suarsa, 2016
13	1408,82	C=C Aromatik (1400-1500)	Tiwow et al., 2021
14	1260,90	R-O-R Eter (1000-1300)	Prasetyo et al., 2023
15	1043,33		
16	778,37	C-Cl Chloride (540-785)	Nurhidayati et al., 2021
17	687,90		

At a wavelength of 3860.26-3621.87 cm-1, it shows that there is a hydroxyl O-H functional group in activated micro biochar. According to Zhang et al. (2020), the hydroxyl OH group has a wavelength range between 3300-3800 cm-1. In addition, there is also a carboxyl C=O group at peaks of 1738.41 cm-1 and 1693.89 cm-1. The presence of negatively charged groups such as hydroxyl and carboxyl in activated carbon shows that activated carbon can function to bind cations, especially cations with high valence such as Al3+ and Fe3+ or other heavy metal compounds (Santi and Goenadi, 2012). The carboxyl group is a typical functional group found in activated carbon and indicates that an active carbon substance has been formed (Mentari et al., 2018). According to Purwani (2023), the more functional groups such as hydroxyl and carboxyl, the more pollutants can be adsorbed by the adsorbent, in this case activated carbon, so that it can increase its adsorption capacity. The FTIR curve also forms a triple carbon C≡C functional group with three peaks, namely 2376.04 cm-1, 2311.42 cm-1, and 2234.59 cm-1. This triple carbon group indicates an increase in carbon content (Mentari et al., 2018). According to Nasution and Rambe (2013), the presence of the C≡C and C=C groups indicates that the C or carbon content in biochar is purer. In addition, the FTIR results also contain an N=O group at a wavelength of 1522.99 cm-1. These results indicate that activated carbon contains a nitro group (Mentari et al., 2018). FTIR results show the presence of C-Cl chloride functional groups at peak wavelengths of 778.37 cm-1 and 687.90 cm-1. According to Wijaya et al. (2020), the C-Cl functional group plays a role in the absorption of mercury (Hg) metal.

Conclusion

The conclusion of this study is: 1). The effect of HCl activator concentration and soaking time has been evaluated using the characteristics of moisture content, ash content, volatile matter content, fixed carbon content, and iodine number. The activating concentration factor and soaking time through statistical analysis were proven to influence all the parameters tested. These results are proven by the results of samples after activation which have better quality than control samples without activation. It was found that the result tended to be that the higher the concentration factor used and the longer the soaking time, the lower the moisture content, ash content and volatile matter content. Inversely proportional to the bound carbon content and iodine number which increases with increasing activator concentration and soaking time. 2). The results of determining the best treatment using the Multiple Attribute Zeleny method showed that the treatment concentration that tends to be 2M HCl concentration with a soaking time of 24 hours is the best treatment. This treatment has met two of the five SNI standards, which have characteristics of water content of 1.20%, volatile matter content of 11.65%, ash content of 48.62%, bound carbon content of 40.66%, iodine absorption capacity of 470.98 mg/g and a yield of 83.19%. This best treatment was also tested using PSA and the particle size was 2.284 μm, which proves that the particles are micro-sized.

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