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PENGARUH KONSENTRASI LARUTAN PENGIMPREGNASI TERHADAP KARAKTERISTIK KARBON AKTIF

THE EFFECT OF IMPREGNATING SOLUTION CONCENTRATION ON THE CHARACTERISTIC OF ACTIVATED CARBON

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ABSTRAK

Sebagai limbah pertanian, sekam padi dapat diproduksi sebagai karbon aktif karena kandungan selulosa yang tinggi. Penelitian ini bertujuan untuk mengetahui karakteristik karbon aktif yang terimpregnasi larutan seng klorida ($ZnCl_2$) dengan variasi konsentrasi. Karbon aktif dibuat dari sekam padi dengan impregnasi $ZnCl_2$ menggunakan metode perendaman. Perendaman dilakukan selama 24 jam dengan impregnasi $ZnCl_2$ pada berbagai konsentrasi 10%, 20%, dan 30%. Dilakukan analisis proksimat terhadap ketiga variasi konsentrasi karbon aktif. Karbon aktif terbaik diperoleh dengan impregnasi $ZnCl_2$ 30%, dimana kadar air 5.08%, kadar abu 24.58%, kadar bahan yang mudah menguap 25.19%, dan kadar karbon terikat 50.23%. Bilangan Iodium dan jumlah adsorpsi metilen biru diperoleh pada konsentrasi maksimum 30% $ZnCl_2$ yaitu 631 mg/g dan 322 ml/g. Uji karakterisasi karbon aktif yang diimpregnasi menggunakan FTIR telah dilakukan. Gugus fungsional ditemukan dalam karbon aktif yang diimpregnasi seng klorida, terdiri dari vibrasi gugus OH hidroksil pada 3200-3600 cm^{-1}, vibrasi C=C aromatik pada 1400-1600 cm^{-1}, vibrasi C=O pada 2300 cm^{-1}, dan vibrasi C-O pada 1200 cm^{-1}. Sementara itu, impregnasi pada bilangan gelombang 1026 cm^{-1} dimungkinkan merupakan SiO_2.

ABSTRACT

As an agricultural waste, rice husk is able to be produced as activated carbon due to its high cellulose content. This research aimed to determine the characteristics of activated carbon impregnated with zinc chloride ($ZnCl_2$) solution at various concentrations. The activated carbon prepared from rice husk with $ZnCl_2$ impregnation was investigated under soaking treatment. Soaking was carried out for 24 hours with $ZnCl_2$ impregnation at various concentrations 10%, 20%, and 30%. Proximate analysis was performed on the three variations of activated carbon concentration. The best activated carbon was obtained by 30% ZnCl2 impregnation, where the moisture content was 5.08%, the ash content was 24.58%, the volatile matter content was 25.19%, and the fixed carbon content was 50.23%. Iodine number and amount of methylene blue adsorption were obtained at the maximum concentration of 30% $ZnCl_2$ which are 631 mg/g and 322 ml/g. Impregnated activated carbon characterization test with FTIR has been carried out. The functional group is found in zinc chloride-impregnated activated carbon, consisting of OH stretching of hydroxyl groups at 3200-3600 cm^{-1}, aromatic C=C vibrations at 1400-1600 cm^{-1}, C=O vibration at 2300 cm^{-1}, and C-O vibration at 1200 cm^{-1}. Meanwhile, the absorption in band 1026 cm^{-1} corresponds to SiO_2.

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INTRODUCTION

One of the low-cost and green alternative materials produced as activated carbon is rice husk. It is resulted from rice milling and contains about 20% of the paddy weight (Menya, et al., 2018). It is available as one of the most abundant, renewable, and inexpensive carbon sources. Rice husk is a very potent source of porous carbon material due to its 50% cellulose content (Sukasri, et al., 2021). Activated carbon has been widely used as an adsorbent, especially in water treatment processes because of its large surface area, high microporosity, and adsorption capacity (Muniandy, et al., 2014).

The existence of rice husks in Indonesia is quite abundant, especially in South Sulawesi Province. Based on Statistics Indonesia (BPS) data, rice productions in South Sulawesi reach 5.15 million tonnes in 2021 and subsequently result in rice husks of around 15% to 20%. Based on this data, it can be calculated that the rice husk production can reach 1.03 million tonnes per year. Commonly, rice husks are just utilized as a direct fuel. The remains are not utilized and might be the potential to pollute the environment. An alternative solution offered is to make it into activated carbon to reduce the environmental harming effect.

Activated carbon is derived from rice husks as a result of rice mills. The basic methods for preparing activated carbon include physical and chemical activation (Li, et al., 2015). Chemical activation is more beneficial than physical activation because chemical activation will produce both a higher carbon yield and a better-developed pore structure. Pyrolysis of rice husks is carried out on physical activation, followed by gas activation such as steam or CO_2 at temperatures above 600 °C (Liu, et al., 2010). However, in chemical activation, activating substances such as H_3PO_4, KOH, or $ZnCl_2$ are used to impregnate rice husks. During the activation process, functional groups will form on the porous carbon surface and this surface will be reactive, thereby affecting its adsorption properties (Sukasri, et al., 2022).

Several researchers use the carbonization process in the manufacture of porous carbon such as (Zhang, et al., 2017), (Hanum et al., 2017), and (Hendrawan, et al., 2019) because carbonization can release volatile substances and decompose organic matter in rice husks. However, too-high temperatures can close the pores of the activated carbon due to the formation of large amounts of ash.

Figure 1. Rice Husk

This research aims to impregnate porous carbon with $ZnCl_2$ with various concentrations to produce activated carbon, to analyze the characteristics of impregnated activated carbon including moisture content, ash content, volatile

matter content, fixed carbon content, iodine number, and methylene blue number. The activated carbon was then analyzed by Spectrophotometer FTIR.

METHOD

The materials used in this study were rice husk, zinc chloride, distilled water, iodine solution, and $Na_2S_2O_3$ solution. Rice husks were collected from rice mills in Maros Regency, South Sulawesi. Zinc chloride and sodium thiosulfate were purchased from Merck. Variations of zinc chloride concentration were made, 10%, 20%, and 30% (w/v).

The apparatus in this study consisted of analytical balance, porcelain cup, chemical glassware (Pyrex), magnetic stirrer, aluminum foil, desiccator, oven (Ecocell), and FTIR (Shimadzu 8400S).

Preparation of Activated Carbon

Rice husks were washed repeatedly using distilled water to remove some impurities, dried in the sun for a day, and then burned for 40 minutes until they become charcoal. The carbon obtained from the combustion was then cooled, crushed, and sieved with a size of 60 mesh. The carbon that passes through the sieve was ready for the impregnation stage.

Impregnation Process

A total of 10 g porous carbon were impregnated with 100 mL $ZnCl_2$ 10%, 20%, and 30% (w/v). The impregnation process using the maceration method was carried out by soaking the porous carbon with zinc chloride for 24 hours at room temperature. After 24 hours, the samples were filtered through filter paper to separate filtrate and residue. The residue was washed with distilled water three times until neutral (pH = 7) then dried in an oven at a temperature of 105 °C until a constant weight was obtained. Then, activated carbon was analyzed with FTIR.

Absorption of Iodine

A total of 0.5 g of activated carbon was added to 25 mL of 0.1 N iodine solution. The solution was shaken for 15 minutes and then filtered. The filtrate obtained was taken at 10 mL and titrated with 1 N $Na_2S_2O_3$ solution.

Adsorption of Methylene Blue

Buffer solution and methylene blue solution were prepared. An amount of 0.1 g of activated carbon was put into Erlenmeyer 500 mL and then titrated with methylene blue solution while shaken occasionally. The titration was stopped when the color of the solution was the same.

Proximate Analysis (SNI No.06-3730-1995)

Characterization was carried out to find out the basic properties of activated carbon including moisture content, ash content, volatile matter content, and fixed carbon content. The standard activated carbon quality we used refers to SNI 06-3730-1995 regarding technical activated carbon as presented in Table 1.

Moisture content: 1 g sample was placed in a porcelain cup and heated at 110°C for 3 hours. Then the sample was cooled in a desiccator and the dry sample weight was measured. Ash content: 1 g sample was placed in a porcelain cup and heated in a furnace at 800 °C for 2 hours. Then cooled in a desiccator, and weighed. Volatile matter: 1 g sample was placed in a porcelain cup and heated in a furnace at 950°C for 5 minutes. Then cooled in a desiccator, and weighed. Fixed carbon: Fixed carbon content is obtained by reducing 100% with the sum of the ash content and volatile matter content.

Data Analysis

Moisture content was calculated as follows:

$$M = \frac{(w1-w2)}{w2} \times 100\% \quad (1)$$

Remark:
M = moisture content
w_1 = weight of the initial sample
w_2 = weight of the sample after heating

Ash content was calculated as follows:

$$A = \frac{w1}{w2}\ x\ 100\% \quad (2)$$

Remark:
A = ash content
w_1 = weight of residual incandescent
w_2 = weight of simple

Volatile matter content was calculated as follows:

$$Vm = \frac{[100(w1-w2)-M(w1-w)]}{(w1-w)(100-M)} \times 100\% \quad (3)$$

Remark:
Vm = volatile matter content
w_1 = weight of porcelain, lid, and initial sample
w_2 = weight of porcelain, lid, and sample after heating
w = weight of empty porcelain and lid
M = % of moisture determined above

Fixed carbon content was calculated as follows:

$$FC = 100\% - (\%A + \%Vm) \quad (4)$$

Remark:
FC = Fixed carbon content
%A = % ash content
%Vm = % Volatile matter content

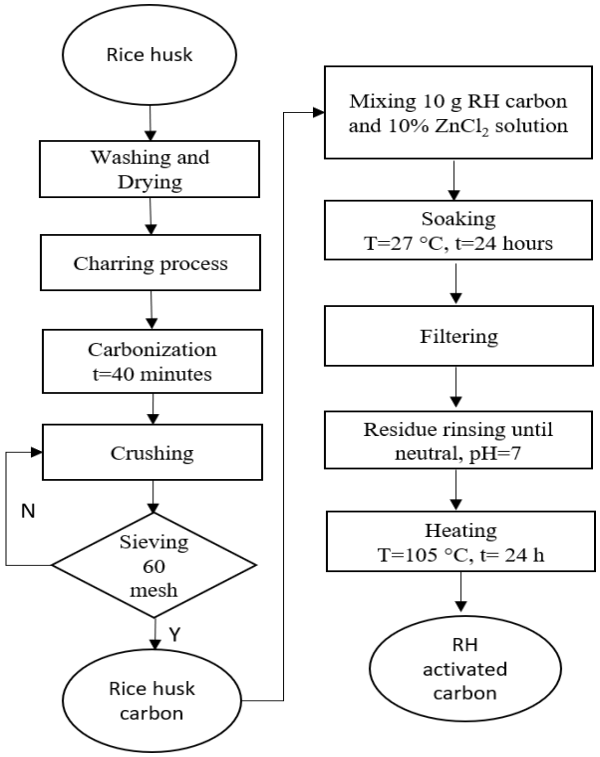

Figure 2. Carbonization and Rice Husk Activation Process

Fourier Transform Infrared (FTIR) Spectroscopy

Functional groups of rice husk-activated carbon analyzed and characterized by FTIR. In this research, FTIR spectroscopy from the Shimazu brand was used. FTIR analysis was carried out by mixing activated carbon with KBr to form pellets and then scanning from 4000 to 500 cm^{-1}.

RESULT AND DISCUSSION

The standard specification for activated carbon according to SNI No.06-3730-1995 was shown in Table 1.

Table 1. Standard Specification for Activated Carbon

Analysis	Unit	Quality Requirements	
		Particle	Powder
Volatile matter 950 °C, %	-	Max. 15	Max. 25
Moisture content, %	-	Max. 4,4	Max. 15
Ash content, %	-	Max. 2,5	Max. 10
Parts that are not carbonized	-	0	0
Iodine number	mg/g	Min. 750	Min.750
Pure activated carbon, %	-	Min. 80	Min. 65
Benzene adsorption capacity, %	-	Min. 25	-
Methylene blue number,	mg/g	Min. 60	Min. 120
Bulk specific gravity	g/ml	0,45-0,55	0,3-0,35
Escaped mesh 325%	-	-	Min. 90
Distance, %	-	90	-
Violence, %	-	80	-

Whereas Table 2 showed the result of activated carbon which was impregnated with $ZnCl_2$ 10%, $ZnCl_2$ 20%, and $ZnCl_2$ 30% using the maceration method. We could see the result of the proximate analysis of activated carbon from rice husk including moisture content, ash content, volatile matter content, and fixed carbon content in Table 2. The result of the iodine number and methylene blue number is also given.

Table 2. Results of Chemical Composition Analysis of $ZnCl_2$ Impregnated Activated Carbon

Activated carbon analysis	Results based on impregnation solution		
	$ZnCl_2$ 10%	$ZnCl_2$ 20%	$ZnCl_3$ 30%
Moisture content	5.16%	5.00%	5.08%
Ash content	42.56%	39.97%	24.58%
Volatile matter content	27.08%	26.97%	25.19%
Fixed Carbon content	30.36%	33.06%	50.23%
Iodine number	606 mg/g	619 mg/g	631 mg/g
Methylene blue number	300 mg/g	300 mg/g	322 mg/g

Figure 3. Impregnation process of carbon with $ZnCl_2$

The moisture content of activated carbon obtained was in the range of 5% either impregnated with $ZnCl_2$ with different concentrations. This result still met the SNI standards where the moisture content of activated carbon is below 15%. This shows that the activated carbon surface contains a few polar functional groups. Low moisture content indicates that a small amount of water is left behind and covers the pores of the activated carbon (Kusdarini, et al., 2017).

Based on Table 2, it could be observed that increasing $ZnCl_2$ concentration during impregnation, caused ash content to decrease. Although it has

decreased, we could see that the ash content far exceeded the SNI requirements. The cause of the high levels of ash is possible due to the process of oxidation of carbon. The carbonization process produces oxygen from burning organic molecules (Arneli, et al., 2017).

Volatile substances were produced from the carbonization process. The high carbonization temperature would evaporate the volatile compounds and would increase the number of pores formed. Based on our research, volatile matter content did not meet the requirements of SNI where the maximum volatile matter levels were 25%. From Table 2, increasing the concentration of Zinc chloride as an impregnation solution could reduce the volatile matter content. Volatile substances will be easily released from the activated carbon surface during the activation process with increasing activator concentrations (Heidarinejad, et al., 2020).

The amount of carbon contained in activated carbon is indicated by the fixed carbon content. According to (Febryanti, et al., 2015), the fixed carbon content is affected by the ash content, the volatile matter content, the cellulose, and the lignin content which can be converted into carbon atoms. The highest fixed carbon content was obtained from activated carbon impregnated with 30% Zinc chloride. This result did not meet the SNI standard where the fixed carbon content is at least 65%. Fixed carbon content also correlated to the concentration of impregnation solution. The increase of activator concentration was followed by the increase in fixed carbon content (Maulina, et al., 2020).

One indicator of the absorption ability of activated carbon is the iodine number. In this study, activated carbons have capacities in the range of 606 mg/g to 631 mg/g. The iodine number tends to increase after the increasing concentration of the impregnation solution. The highest iodine number is 631 mg/g at 30% Zinc chloride. In line with research conducted by (Machrouhi, et al., 2019) who state that the impregnation solution ratio has a significant effect on the iodine number. The high iodine number will lead to a higher number of micropores. The level of the iodine number is also affected by the ash content. High ash content will cause a low iodine number because the ash will clog the pores so the absorption of activated carbon decreases (Oko, et al., 2021).

Methylene blue adsorption capacity with various concentrations of $ZnCl_2$ impregnation complied with SNI standards. The maximum adsorption capacity for methylene blue is 322 mg/g. However, this result is lower than previous research (Zhao, et al., 2022) which obtained the maximum absorption capacity of methylene blue of 869.57 mg/g from activated carbon with $ZnCl_2$. As reported by (Yahya et al., 2018) in a previous study that the porosity and functional group composition in activated carbon with Zinc chloride chemical treatment had better methylene blue adsorption. From this study, there was no significant difference in the absorption capacity of methylene blue with various concentrations of Zinc chloride. The high adsorption capacity indicates that the pores of the activated carbon are getting bigger. This will affect the ability of activated carbon in metal adsorption which is also higher.

FTIR Analysis of Activated Carbon

Activated carbon which has been impregnated with Zinc chloride with various concentrations was analyzed for its functional groups using infrared spectroscopy. From Figure 4, it can be seen that the three FTIR results from zinc chloride-impregnated activated carbon tend to be almost similar, which indicates that

activated carbon contains identical functional groups. Stretching vibrations of the hydroxyl groups (-OH) are found in several peaks in the wavenumber region between 3200-3600 cm^{-1} (Wazir, et al., 2020). This is due to the presence of increased cellulose and decreased part of hemicellulose.

Aliphatic CH absorption from activated carbon can be found in wave numbers around 2800-2900 cm^{-1}. A fairly sharp absorption at wave number 2300 cm^{-1} indicates the presence of C=O group vibrations (Tiwow, et al., 2021).

The peak observed at 1700 cm^{-1} is the stretching vibration of carbonyl/carboxyl groups, whereas C=C stretch aromatics are found in the wave number at 1400-1600 cm^{-1} (Kaya, et al., 2018). C-O stretching vibration can be observed at sharp wavenumbers around 1200 cm^{-1}. The possibility of the presence of silica is found in absorption at wave numbers 1026 cm-1, 780-790 cm-1, and at wave number 447 cm^{-1} (Menya, et al., 2020).

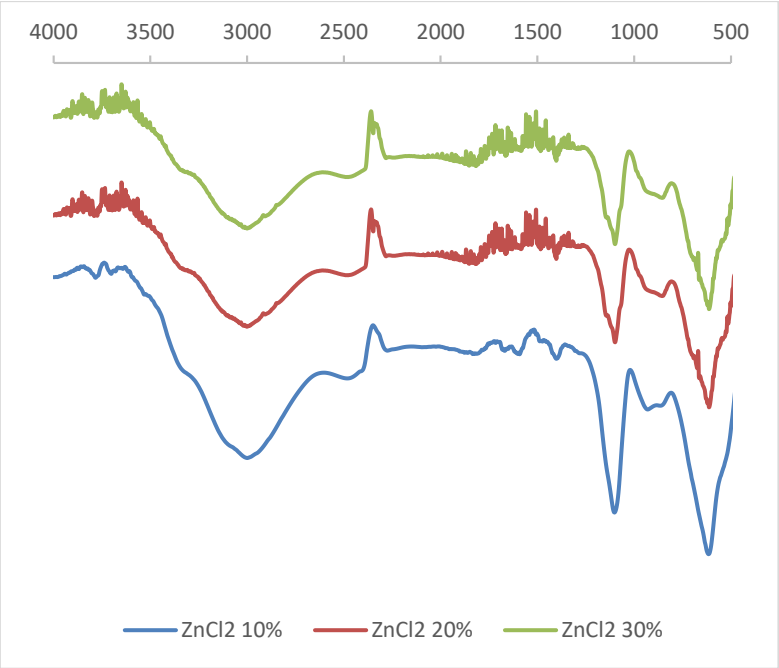

Figure 4. FTIR spectra of activated carbon

CONCLUSIONS

Activated carbon impregnated with zinc chloride 30% was the best-activated carbon in the study, which had a moisture content of 5.08%, ash content of 24.58%, volatile matter content of 25.19%, fixed carbon content of 50.23%, iodine number 631 mg/g, and methylene blue number 322 mg/g. The functional group is found in zinc chloride-impregnated activated carbon, consisting of OH stretching of hydroxyl groups at 3200-3600 cm^{-1}, aromatic C=C vibrations at 1400-1600 cm^{-1}, C=O vibration at 2300 cm^{-1}, and C-O vibration at 1200 cm^{-1}. Meanwhile, the absorption in band 1026 cm^{-1} corresponds to SiO_2. We hope further research will add the characterization of the analysis with a surface area analyzer to determine the surface area of the pores.

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