CHARACTERIZATION OF SALAK WEDI ACTIVATED CARBON STRUCTURE USING KOH AND ZnCl₂ ACTIVATOR

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ABSTRACT

Activated carbon can be made from a variety of organic and inorganic raw materials. One of the organic materials with great potential in the manufacture of activated carbon is the bark of salak wedi (Salacca Zalacca). The purpose of this study was to determine the physical characteristics of the activated carbon of salak wedi bark which has been chemically activated using KOH and ZnCl₂ with the process of being activated once using only KOH or ZnCl₂ and multilevel activation using a combination of KOH and ZnCl₂ to determine the number of pores in the sample and to determine the presence of a crystal structure in the sample. The research method includes sample making, single and multilevel activation have more pores than the activated carbon sample of bark once. The SEM results obtained that the multilevel activation has a larger number of pores compared to the activated carbon + ZnCl₂ (s) is 35,976,798,714 nm3. The XRD analysis indicated that activated carbon from the bark of salak wedi is a material with good conductivity properties because it has a relatively high crystalline structure, both on one-time activation and multilevel activation due to be activation.

Keywords: Carbon active, Salak, KOH and ZnCl₂, Characterization

INTRODUCTION

The manufacture and utilization of activated carbon from biomass, especially as a supercapacitor electrode application has been introduced for the benefit of biomass, energy, and future energy development [1]. Activated carbon from biomass can be made from bark, corn cobs, bagasse, coconut shells, rice husks, sawdust, hardwood, and coal [2]. Salak wedi bark is one of the interesting biomass wastes to be studied as a raw material for making activated carbon because it is the largest part of salak waste that is rarely used. In Bojonegoro, salak fruit is a plant that is very easy to get because there is a salak-producing area, namely Wedi. There usually only use the fruit to be processed into food, which leaves so much bark waste.

The manufacture of activated carbon consists of two stages, namely the process of carbonization and activation. Carbonization is a process of burning raw materials at high temperatures which causes decomposition of organic compounds that make up the structure of raw materials [3], while the chemical activation process aims to increase the surface area and porosity of activated carbon [4]. In addition to the large surface area, surface modification also has an influence on the characteristics of a carbon material, especially as supercapacitor electrode material [5]. This а modification can be a chemical or physical activation. Modification of the activated carbon surface aims to create functional groups on the carbon surface which are

reported to have an effect on increasing the capacitance value of activated carbon as an electrode material [6]

Modified the surface of activated carbon from cassava bark using a solution of HNO_3 , $H2SO_4$, and $H2O_2$ researched by Ismanto [7]. The results showed that the highest specific capacitance value was obtained from activated carbon whose surface was modified with HNO3, 72.6% which was higher than activated carbon without surface modification.

In this study, surface modification of activated carbon using bark waste was carried out using chemical activation in stages using KOH and ZnCl₂. The activated carbon that has been activated will then be characterized using SEM to determine the number of pores in the sample and an XRD test is also carried out to determine the presence of a crystal structure in the sample.

METHODS

a. Materials

The biomass used in this research is bark waste of salak wedi. The skin of the salak fruit is cleaned with water until clean and then dried in an oven at 110° C to remove moisture in the washing. The activation stage is chemical activation using ZnCl₂ and KOH activators.

b. Method and Procedure

The salak wedi bark was carbonized in a furnace at 500°C for 2 hours, then allowed to stand in a desiccator, pulverized and then sieved using a 100 mesh sieve. The next step is activated carbon powder with activators (KOH and ZnCl₂) with a powder and activator ratio of 1:4. Previously, a solution of activator and distilled water (with a concentration of 20%) was made, then carbon powder was added according to the ratio and stirred with a magnetic stirrer for 20 hours. Then the samples were dried and washed with distilled water + dilute HCl to obtain a pH of 6-7[8]. The last stage was dried again in the oven to obtain modified activated carbon samples. The third variation is the result of activated carbon ZnCl₂ being reactivated using a KOH activator (ZnCl₂ + KOH(s)), and the fourth variation is the result of KOH carbon being reactivated using a ZnCl₂ activator (Carbon $KOH + ZnCl_2(s)).$

c. Sample Test

The samples obtained were then tested for the characterization of their physical properties using a scanning electron microscope to determine the microstructure of the activated carbon of the bark of the bark. In addition, an XRD test was carried out to determine the presence of a crystalline structure in the sample.

RESULT AND DISCUSSION

a. SEM Test Result

The surface morphology of salak wedi bark activated carbon with a one-time activator with graded was characterized using SEM, and the results are shown in Figure 1. Figure 1 shows the appearance of pores in all activations. The pores formed indicate that the breakdown process of organic compounds has occurred, and it appears that the pore diameters are not homogeneous. It is thought to be due to the activation process that does not flow with nitrogen gas [9], so the pores formed cannot be uniform.

Figure 1 shows the pore size of activated carbon activated once was smaller than that of activated carbon that was activated twice. Meanwhile, in the stratified activation of KOH + $ZnCl_2$ (s) Carbon, it can be seen that the pore size produced is the largest than the others. Calculations were carried out using the ImageJ program to determine the number of pores of activated carbon produced, which is presented in the graph below.

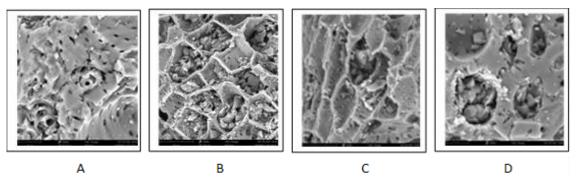
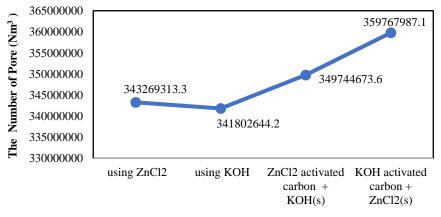


Figure 1. SEM results carbon powder (a) activated using ZnCl₂ (b) activated using KOH (c) ZnCl₂ activated Carbon + KOH(s) (d)KOH activated Carbon + ZnCl₂(s)

Based on this graph figure 2, it can be seen that with the stratified activator variations. The pore volume in the activated carbon sample which was activated twice had a higher pore volume value. The value of the pore volume on the variation of Carbon $ZnCl_2 + KOH$ (s) is $34,974,467,360 \text{ nm}^3$ and on the variation of Carbon KOH + ZnCl₂ (s) of $35,976,798,714 \text{ nm}^3$. With the stratified variation in the activator, more hydrocarbons or impurities are lifted, increasing the pore volume of the activated carbon.



Sample Of Activated Carbon

Figure 2. Graph the number of pores of activated carbon

This increase in pore volume results from dissolved impurities that fill the pore system of the material during the activation process twice (tiered). In addition, the activation process of activated carbon with acid serves to dissolve the impurities that arise during the carbonization process that covers the pore system of the activated carbon[10].

The results of SEM with multilevel activation have more pores than the activated carbon sample of bark once. However, the pore distribution of these four samples is still uneven. This may also be due to the nonflowing nitrogen gas during the carbonization process which also affects the uneven distribution of pores on the surface of the activated carbon of the bark [11]. In addition, the SEM results obtained that the multilevel activation has a larger number of pores compared to the activation which is only carried out once, with the result that the largest pore volume with the stratified activator Carbon KOH + $ZnCl_2(s)$ is 35,976,798,714 nm³.

b. XRD test results

The XRD diffraction pattern of a sample of salak wedi bark carbon powder is shown in Figure 3.

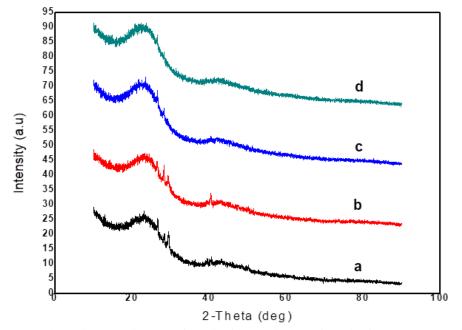


Figure 3. XRD results carbon powder (a) activated using ZnCl₂ (b) activated using KOH (c)ZnCl₂ activated Carbon + KOH(s) (d)KOH activated Carbon + ZnCl₂(s)

In Figure 3 it can be see that from all the results of the diffraction on the carbon powder of salak wedi bark, it was shown that an amorphous structure was formed with widening and increasing peaks. The increasing of this diffraction peak indicates that the degree of crystallinity is also getting higher. Structure micro carbon active formed is structure tagged turbostratic with description structure crystals that are not in order that is existence major and minor peaks. On carbon active skin snake fruit, this could see peak the highest of each variation activation at a 2 θ angle of about 26° to 30° and a peak other about 39° to 41°. High and low the resulting peak from carbon XRD characterization active skin snake fruit this affected by the activation process that causes happening shift plate the original hexagonal level regularity high (crystalline) to no regular (amorphous) appropriate with research conducted by [12]. The bond hexagonal is shaped bond aspect six on the surface carbon normal active called pores. Based on pattern diffraction, carbon x-ray active obtained, then analyzed degrees crystallization to obtain comparison degrees crystalline carbon using Origin software in Table 1.

Phase	Activated carbon using ZnCl ₂	Activated carbon using KOH	ZnCl ₂ Activated Carbon + KOH(s)	KOH Activated Carbon + ZnCl ₂ (s)
	%	%	%	%
Crystalline	39.88%	45.66%	31.86%	33.83%
Amorphous	60.12%	54.34%	68.14%	66.17%

Table 1. Comparison Degrees Crystalline Carbon

Based on Table 1., can we see more significant changes in carbon activated once using the KOH activator, where phase crystalline gets the most significant percentage compared with another activation. High and low, the resulting peak from XRD characterization is affected by the activation process chemistry done with using ZnCl₂ and KOH activator which causes shift plate the original hexagonal level regularity [13]. Research results follow the theory proposed by Fagbohun,E.O., that carbon active with more KOH activator could react with carbon so that ingredient standard with more carbon tall more good use KOH activator [14]. That thing causes degrees of crystallinity to increase. The results of the degree of crystallinity in this study using activated carbon from the bark of salak wedi are higher than the degree of crystallinity of activated carbon synthesized from other biomass materials such as coconut fiber [15,16].

CONCLUSION

This study found that the XRD analysis indicated that activated carbon from the bark of salak wedi is a material with good conductivity properties because it has a relatively high crystalline structure, both on one-time activation and multilevel activation. Time using KOH activator obtained phase crystalline with the most significant percentage if compared with another activation. In addition, the SEM results obtained that the multilevel activation has a larger number of pores and porosity levels when compared to the activation, which was only carried out once, with the result that the largest pore volume with the stratified KOH activated Carbon + $ZnCl_2$ (s) was 35,976,798,714 nm³.

REFERENCES

- Kalyani, P. and Anitha, A., 2013. Biomass carbon & its prospects in electrochemical energy systems. *Int J Hydrogen Energy*. Vol. 38 (10) pp. 4034– 4045.
- [2] Mdoe, J. E., 2014. Agricultural waste as raw materials for the production of activated carbon. *Huria: journal of the open university of Tanzania*. Vol. 16 pp. 89–103.
- [3] Zhang, X., Zhang, L., and Li, A. 2019. Cohydrothermal carbonization of lignocellulosic biomass and waste polyvinyl chloride for highquality solid fuel production: Hydrochar properties and its combustion and pyrolysis behaviors. *Bioresour Technol.* Vol. 294 pp. 122113.
- [4] Sayğl, H. and Güzel, F., 2016. High surface area mesoporous activated carbon from tomato processing solid waste by zinc chloride activation: process optimization, characterization and dyes adsorption. J Clean Prod. Vol. 113, pp. 995–1004.
- [5] Mehdi, R., Khoja, A. H., Naqvi, S. R., Gao, N., & Amin, N. A. S, 2022. A Review on Production and Surface Modifications of Biochar Materials via Biomass Pyrolysis Process for Supercapacitor Applications. *Catalysts.* vol. 12 (7) pp. 798.
- [6] Abioye, A. M. and Ani, F. N., 2015. Recent development in the production of activated carbon electrodes from agricultural waste biomass for supercapacitors: A review. *Renewable and sustainable energy reviews*. Vol. 52 pp. 1282– 1293.
- [7] Ismanto, A. E., Wang, S., Soetaredjo, F. E. and Ismadji, S., 2010. Preparation of Capacitor's

Electrode from Cassava Peel Waste. *Bioresource Technology*. Vol. 101 (10) pp. 3534–3540.

- [8] Şahin, Ö., Saka, C., Ceyhan, A. A. and Baytar O., 2015. Preparation of High Surface Area Activated Carbon from Elaeagnus angustifolia Seeds by Chemical Activation with ZnCl₂ in One-Step Treatment and its Iodine Adsorption. *Separation Science and Technology*. Vol. 50 (6) pp.886-891.
- [9] Nurdiansah, H. and Susanti, D., 2013. Pengaruh variasi temperatur karbonisasi dan temperatur aktivasi fisika dari elektroda karbon aktif tempurung kelapa dan tempurung Kluwak terhadap Nilai Kapasitansi Electric Double Layer Capacitor (EDLC). Jurnal Teknik ITS. Vol. 2 (1) pp. F13–F18.
- [10] Permata, A. N., Permatasari, R. R. A. P. and Takwanto, A., 2019. Studi Awal Pengaruh Suhu dan Konsentrasi Pada Proses Aktivasi Karbon dari Kayu Halaban Menggunakan ZnCl2 dan KOH. *DISTILAT: JURNAL TEKNOLOGI SEPARASI*, Vol. 5 (2) pp. 141–146.
- [11] Waluyo, H. M., Faryuni, I. D. and Muid, A., 2017. Analisis Pengaruh Ukuran Pori Terhadap Sifat Listrik Karbon Aktif Dari Limbah Tandan Sawit Pada Prototipe Baterai. Jurnal Fisika Flux: Jurnal Ilmiah Fisika FMIPA Universitas Lambung Mangkurat. Vol. 14 (1) pp. 27–33.
- [12] Tanaka, K., Aoki, H., Ago, H., Yamabe, T. and Okahara, K., 1997. Interlayer interaction of two graphene sheets as a model of double-layer carbon nanotubes. *Carbon N Y.* Vol. (1) pp. 121–125.
- [13] Wulandari, R., Zakir, M., dan Karim, A., 2017. Penentuan Kapasitansi Spesifik Karbon Aktif Tempurung Kemiri (*Alleurites mollucana*) Hasil Modifikasi Dengan HNO₃, H₂SO₄, dan H₂O₂ Menggunakan Metode Cyclic Voltammetry.
- [14] Fagbohun, E. O., Wang, Q., Spessato, L., Zheng, Y., Li, W., Olatoye, A. G., & Cui, Y., 2022. Physicochemical regeneration of industrial spent activated carbons using a green activating agent and their adsorption for methyl orange. *Surfaces and Interfaces*. Vol. 29 pp. 101696.
- [15] Fauziah, N., 2009. Pembuatan arang aktif secara langsung dari kulit acacia mangium wild dengan aktivasi fisika dan aplikasinya sebagai adsorben, UT – Forest
- [16] Hoang, A. T., Kumar, S., Lichtfouse, E., Cheng, C. K., Varma, R. S., Senthilkumar, N., Nguyen, P. Q. P. and Nguyen, X. P., 2022. Remediation of heavy metal polluted waters using activated carbon from lignocellulosic biomass: An update of recent trends. *Chemosphere*. Vol. 302 pp. 134825