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Production Process of Large Pore Size Activated Carbon from Palm Kernel Shell using Sodium Chloride as An Activator

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Abstract

This study aimed to determine the yield of activated carbon, iodine number, and surface area of palm activated carbon. Samples were produced by using sodium chloride (NaCl) as an activator. Palm shells that had been produced by the milling process were then sieved with a 12 mesh sieve and soaked in 20 % NaCl solution. The sample solution was heated over a water bath at 70 ºC and continued with the drying process at a constant temperature of 105 ºC. The activated shells continued the pyrolysis process at temperatures of 300, 400, and 500 ºC for 3 hours. The activated carbon obtained from the pyrolysis process was weighed and then washed using hot distilled water. The samples were dried in an oven at a temperature of 105 ºC for 24 hours. The results were analyzed for iodine number using iodometric titration method, surface area using Brunauer- Emmett-Teller (BET) method, and pore structure using the Scanning Electron Microscope (SEM) method. The results showed the best yield was 38.13 % obtained at 20% NaCl and a temperature of 400 ºC. The best iodine number was 767.745 mg/g and surface area was 6.790 m2/g, pore volume 4.377 cc/g with pore size 9.781 A.

Keywords: Activated carbon, NaCl, SEM, Iodin number, BET

INTRODUCTION

Indonesia is the largest producer and exporter of palm oil in the world. Statically between 2009-2019, there was an increased area of oil palm plantations, with only 7.9 million hectares in 2009, increasing to 14.5 million hectares in 2019 (Badan Pusat Statistik, 2019). The increase in area and production of palm oil resulted in more waste being produced (Schleicher et al., 2019). One of the abundant solid residues of palm oil production is the palm kernel shell. Currently, palm kernel shells are still underutilized and disposed of by burning.

The palm oil processing process produces solid waste similar to empty fruit bunches (EFB) ranging from 22-25% and oil palm shells ranging from 5-7% of the total weight of fresh fruit bunches (FFB). The solid waste produced has great potential due to its high carbon content of 49.79% (Edmund et al., 2014).

In general, coconut shells are used as fuel in electric generators in palm oil processing plants. Oil palm shell is a biomass waste composed of 53.40% lignin, 6.92% cellulose, and 26.12% hemicellulose. From this composition, oil palm shells have a lower flash point and thermal reactivity than empty fruit bunches (FEB) (Baffour-Awuah et al., 2021). Oil palm shells have the characteristics of a porous particle surface, low sulfur content, high mechanical strength, various functional groups, and insoluble in water (Rashidi & Yusup, 2021).

Based on these compositions and characteristics, activated charcoal is one of the products with high economic value that can be utilized from oil palm shells. Activated charcoal is a highly porous material widely used in the separation process, both in the purification of liquid waste and the separation of gas phases. According to Heidarinejad et al. (2020) activated charcoal is an adsorbent material with a high degree of porosity and surface area and is composed of 90% carbon. Activated carbon is the most popular adsorbent for adsorption because it has a high adsorption capacity (Tanasale et al., 2014). Activated charcoal is a form of charcoal that has been activated by using CO_2 gas, water vapor, or chemicals so that the pore is open. Thus, its adsorption power increases against color and odor substances (Sekewael et al., 2015). The carbon structure of activated charcoal consists of main functional groups such as carbonyl,

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carboxyl, phenol, lactone, and quinone, responsible for the contaminant adsorption process. Activated charcoal also has the advantages of a simpler production process, resistance to corrosion (acid and alkali), and can be used as a supportive catalyst (Rambabu et al., 2015)

Several studies have produced activated charcoal from oil palm shells, including Okoroigwe et al. (2013). Purification of Ubu river water using activated charcoal adsorbents from oil palm shells succeeded in reducing the content of iron compounds from 1.82 mg/l to 0.29 mg/l. Ulfah et al. (2017) reported a study on the recovery of β-carotene compounds from palm oil using activated charcoal from oil palm shells. The adsorption capacity of 14.32 mg/g was obtained with the number of β-carotene compounds recovered was 28-30%. Lee et al. (2021) reported that activated charcoal from oil palm shells could be used in the adsorption process of methylene blue compounds with an adsorption capacity of 16.92 mg/g. Tan et al. (2021) reported that activated charcoal from oil palm shells could be used as an adsorbent in the POME waste treatment process, with a color removal efficiency of 99.7% and COD of 85.0%.

Before being used as activated charcoal, oil palm shells go through two stages, namely carbonization, and activation. This process aims to increase oil palm shell-activated charcoal's porosity and surface area (Andas et al., 2017). There are three activation processes for charcoal: chemical, physical, and chemical-physical combinations. The physical activation process is carried out under atmospheric pressure using gas O_2, CO_2, N_2, and water vapor at a temperature of 800-1100 °C (Heidarinejad et al., 2020). The physical activation process has weaknesses such as long activation time, the low adsorption capacity of the adsorbent, and high energy consumption. The chemical activation process or wet oxidation of activated charcoal raw materials will be mixed with an activating agent, and then the mixture is heated at a temperature of 400-900 °C (Hidayu & Muda, 2016). The chemical activation process has advantages such as a larger surface area of the adsorbent, higher porosity, shorter activation time, and lower energy consumption. The activators that are generally used to make activated charcoal are H_3PO_4, KOH, $ZnCl_2$, NaOH, and K_2CO_3.

Several studies have been carried out on the process of making activated charcoal from oil palm shells with chemical activation methods. Hidayu & Muda (2016) reported research on the manufacture of activated charcoal using a $ZnCl_2$ activator and obtained a yield of 44% with a BET surface area was 1.223 m^2/g. In a different study, Andas et al. (2017) reported making activated charcoal using a KOH activator and obtained a yield of 8.93% with a BET surface area of 994.83 m^2/g. Nicholas et al. (2018) reported the manufacture of palm shell-activated charcoal using the H_3PO_4 activator, and the BET surface area was 1,169 m^2/g.

In a study conducted by Rashidi & Yusup (2021), making activated charcoal from palm oil-petroleum coke shells using a K_2CO_3 activator reported a significant increase in BET surface area on activated charcoal 317.746 m^2/g from oil palm shells of 1.84 m^2/g.

Each activator commonly used in the production of activated charcoal, especially from biomass waste, has its weaknesses. The H_3PO_4 activator in making activated charcoal from lingo-cellulosic material limits the tar that comes out of the charcoal material during the carbonization process, which affects forming activated charcoal pores (Deliyanni, 2019). $ZnCl_2$ activator is a strong dehydrating agent that prevents tar formation during the process and increases the release of impurities. Still, residues from the $ZnCl_2$ activator will harm the environment (Heidarinejad et al., 2020). When used in biomass waste, the KOH activator produces low yields than other activators (Dzigbor & Chimphango, 2019).

One of the activators that can be an alternative in making activated charcoal from palm kernel shell biomass waste is NaCl. NaCl is non-toxic and environmentally friendly, inexpensive, has high thermal conductivity, and is easily available for use in small-scale production processes (Dzigbor & Chimphango, 2019; Maulina et al., 2020). Several studies have been carried out to produce activated charcoal from biomass waste using NaCl activator. Dolas et al. (2011) reported on the manufacture of activated charcoal from pistachio shells biomass waste. NaCl activator showed the best BET surface area of 703.3 m^2/g compared to alkaline activator NaOH of 370.52 m^2/g and acid activator HCl of 353.25 m^2/g. In the study of making activated charcoal from biomass waste *Acacia auriculeaformis* reported by Kra et al. (2019), the NaCl activator produced the largest activated charcoal yield of 48.87% compared to the H_3PO_4 acid activator of 41.81%. BET surface area of the NaCl activator was 395.40 m^2/g, and the iodine value was 380.71 mg/g. Dzigbor & Chimphango (2019) reported making activated charcoal from mango seed biomass waste using NaCl as an activator, the BET surface area of activated charcoal products was 415 m^2/g. According to Zakaria et al. (2021) apart from the type of activator, the variable concentration of activator, pyrolysis temperature, and activation time affect the yield and surface characteristics of the activated charcoal produced.

Oil palm shells have the potential to be developed as a precursor in the manufacture of activated charcoal. This study aims to examine the effect of the chemical activation method using NaCl activator on yield and characteristics of activated charcoal from oil palm shells. The variables used in this study were activator concentration, pyrolysis temperature, and activation time. Activated charcoal produced will be used as an adsorbent in absorbing iron and turbidity in the underground water purification process. This study produced palm kernel shell-activated carbon by chemical activation using NaCl as an activating agent.

This study aims to produce activated carbon and investigate the effect of NaCl as an activating agent on the characteristic of activated carbon from palm kernel shells. The research variable was pyrolysis temperature. The activated carbon obtained was analyzed for iodine number, surface area using Brunauer-Emmett-Teller (BET) method and pore structure using Scanning Electron Microscope (SEM).

METHODOLOGY

Materials and Instrumentals

The material palm kernel shell was obtained from PMKS PT. Bumi Sama Ganda – Kuala Simpang palm oil mill. Sodium Chloride (NaCl, Merck Millipore) was used as the chemical activator, sodium thiosulfate ($Na_2S_2O_3$, Merck Millipore), and potassium iodine (Merck Millipore). Instruments used in the study included a vertical tubular reactor equipped with a panel system, a gas cylinder with a regulator, and a nitrogen hose connected to the pyrolysis reactor.

Methods

Pre-treatment of Palm Kernel Shell

Palm Kernel Shell (PKS) was used as the precursor for the activated carbon. The PKS were washed using water several times to remove dust and dirt. PKS samples were sundried for two days. Then samples were dried in an oven at 80^0C to remove surface moisture. The dry sample was ground and sieved to the size of 12 mesh and placed in a closed container for further use.

Preparation of Activated Carbon

30 g of raw PKS was mixed with 100 ml of 20% NaCl solution. Impregnation was done at 70 ^{o}C for 3 hours, soaking at room temperature for 24 hours.

After impregnation, samples were dried in an oven at a temperature of 105 ^{o}C to obtain a constant weight. The dried samples were then carbonized at 300 ^{o}C, 400 ^{o}C, and 500 ^{o}C for 3 hours. Meanwhile, the samples without activation directly carbonized after sieving into a size of 12 mesh with the same variation of temperatures.

The resulting activated carbon was cleaned using hot water several times. The washed samples were dried in an oven at 100 ^{o}C for 24 hours to remove internal moisture. Samples were weighed and stored in a closed container for further use and analysis.

Data Analysis

The dry sample was weighed, and the yield percentage was calculated using Equation 1.

$$\text{Yield } \% = \frac{Final\ Mass}{Initial\ Mass}\ x\ 100\% \qquad (1)$$

Where Final Mass = Mass of the product; Initial Mass = Mass of precursor used

SEM analysis (Zeiss-Evo-50) was carried out to determine the surface morphology of NaCl-AC. $Na_2S_2O_3$ volumetric titration method based on ASTM D4607 was used to determine the iodine number of NaCl-AC. The NaCl-AC's specific surface area was determined by Brunauer-Emmett-Teller (BET) method (Quanta-chrome Novawin version 11.0).

RESULTS AND DISCUSSION

Influence of pyrolysis temperature on yield of Activation carbon

Pyrolysis temperature is related to activated carbon yield as seen in Figure 1. Figure 1, shows that the yield of activated carbon indicated decreased with an increase in pyrolysis temperature from 300 ^{o}C to 500 ^{o}C. In this study, the best yield of activated carbon was obtained at a pyrolysis temperature of 300 ^{o}C (38.13%). This phenomenon occurs because the pyrolysis process causes the release of more volatile components as the process temperature increase.

Figure 1 also shows that activated carbon yield before pyrolysis of NaCl activation gives slightly better results than without NaCl activation. The activating agent NaCl acts as dehydrating agent inhibiting tar formation, thereby increasing the carbon yield (Rodrfguez-Reinoso & Sepúlveda-Escribano, 2001)

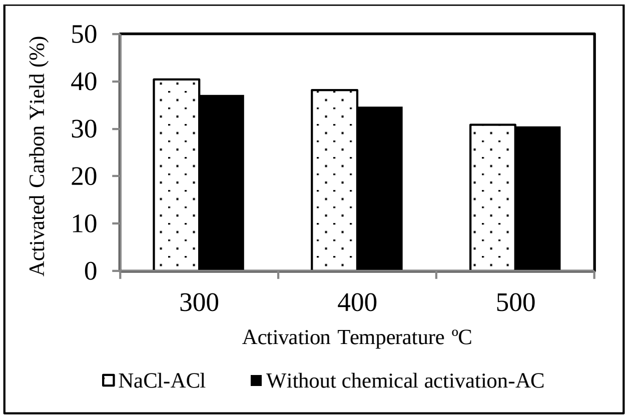

Figure1. Effect of activation temperature on yield Activated Carbon (AC)

Influence of Activation Temperature

The activation temperature of activated carbon yield is also related to the iodine number as seen in Figure 2.

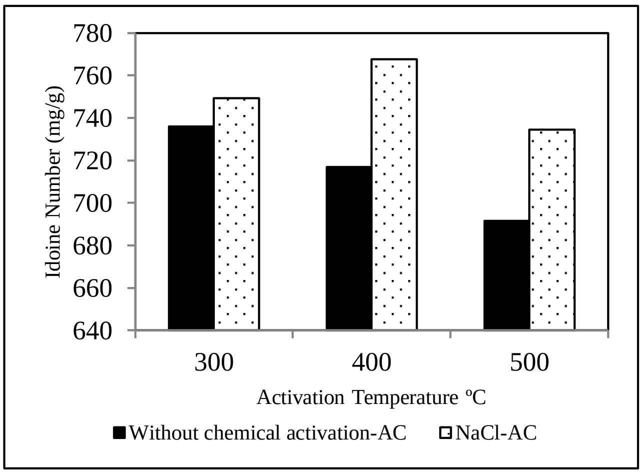

Figure 2. Effect of NaCl on iodine number Activated Carbon (AC)

Figure 2 shows the iodine values of NaCl-AC, which are better than that of AC without chemical activation within the studied temperature range. In this study, the optimum iodine number of activated carbon was obtained at a pyrolysis temperature of 400 ºC with 767.745 mg/g. the increase in pyrolysis temperature and processing time will lead to a decrease in activated charcoal yield.

According to Rodriguez-Reinoso (Rodrfguez-Reinoso & Sepúlveda-Escribano, 2001), the active agent attacks carbon and produces several hydrolysis reactions and causing enhance in the elasticity and swelling of the precursor particles, thus leaving a porous activated carbon when the active agent is removed with distilled water after pyrolysis.

In this study, the increase in iodine number was not linear with increasing temperature. This finding is due to the destruction of the carbon surface due to high-temperature carbonization. Alkali and salt activators tend to produce activated carbon with maximum micro-porous at the carbonization temperature of less than 500 ºC (Maulina et al., 2020)

BET Surface Area

BET analysis was used to show the surface area of activated carbon. BET analysis shows different AC results without activation and AC using NaCl activation in this study. The result presented in Table 1 shows that AC with chemical activation using NaCl provides a larger surface area of 6.790 m2/g than AC without chemical activation of 5.861 m^2/g. NaCl-AC also shows the highest pore volume 4.377 cc/g compare to AC without chemical activation 1.505 cc/g.

This is because an activating agent has contributed to creating more new pores and enlarging existing pores. BET surface area parameter towards activated carbon with and without NaCl as activator is represented in the Table 1.

Table1. BET Surface Area of Carbon

Parameter	AC-Without Chemical Activation	NaCl-AC
Surface area (m^2/g)	5.861	6.790
Pore Volume (cc/g)	1.505	4.377
Pore Size (Å)	9.504	9.781

Morphological Analysis

SEM investigated the surface morphology of AC nonchemical activation and NaCl-AC and the images are shown in Figure 3. Based on Figure 3(a), it can be observed that there was a smooth surface and a small proportion of pores on the particle surface. However, in figure 3(b) there is a lot of development of pores on the particle surface.

The activation treatment aims to enlarge the adsorbent pores (Priyanto et al., 2021). This finding may have resulted from the intercalation of NaCl and the dehydration of volatile materials.

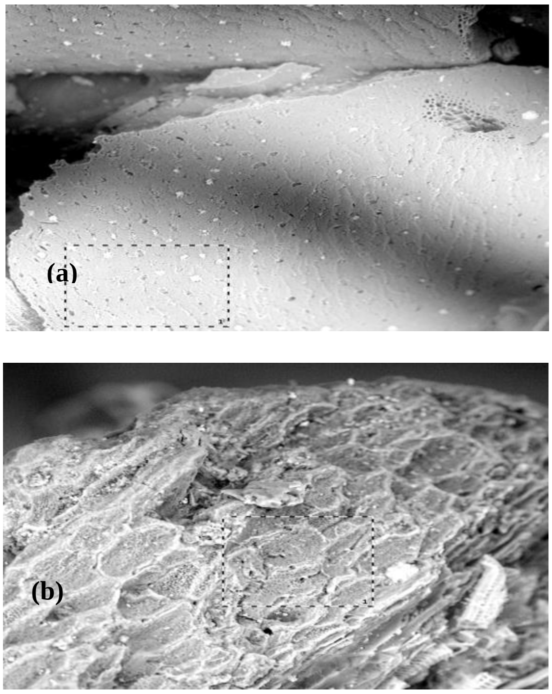

Figure 3. SEM images of (a) AC-without chemical activation and (b) NaCl-AC

CONCLUSION

NaCl can be used as an activating agent to produce activated carbon from palm shells (PKS). This study found the optimum temperature at 400 C with a yield of 38.13% and an iodine value of 767.75 mg/g. activated carbon using NaCl activation has a surface area of 6,791 m^2/g and a pore volume of 4,377 cc/g with a pore size of 9,781 A. NaCl activation in the manufacture of activated carbon from palm shells can be used as an alternative activating agent because it produces many pores on the surface, as shown by SEM Analysis.

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REFERENCES

Andas, J., Rahman, M. L. A., & Yahya, M. S. M. (2017). Preparation and Characterization of Activated Carbon from Palm Kernel Shell. *IOP Conference Series: Materials Science and Engineering*, *226*, 012156.

Badan Pusat Statistik. (2019). *Indonesian Oil Palm Statistic*. BPS-Statistics Indonesia.

Baffour-Awuah, E., Akinlabi, S. A., Jen, T. C., Hassan, S., Okokpujie, I. P., & Ishola, F. (2021). Characteristics of Palm Kernel Shell and Palm Kernel Shell-Polymer Composites: A Review. *IOP Conference Series: Materials Science and Engineering*, *1107*(1), 012090.

Deliyanni, E. A. (2019). Low-cost activated carbon from rice wastes in liquid-phase adsorption. In *Interface Science and Technology*, 30, 101–123.

Dolas, H., Sahin, O., Saka, C., & Demir, H. (2011). A New Method on Producing High Surface Area Activated Carbon: The Effect of Salt on The Surface Area and The Pore Size Distribution of Activated Carbon Prepared from Pistachio Shell. *Chemical Engineering Journal*, *166*(1), 191–197. https://doi.org/10.1016/j.cej.2010.10.061

Dzigbor, A., & Chimphango, A. (2019). Production and Optimization of NaCl-activated Carbon from Mango Seed Using Response Surface Methodology. *Biomass Conversion and Biorefinery*, *9*(2), 421–431.

Edmund, C. O., Christopher, M. S., & Pascal, D. K. (2014). Characterization of Palm Kernel Shell for Materials Reinforcement and Water Treatment. *Journal of Chemical Engineering and Materials Science*, *5*(1), 1–6.

Heidarinejad, Z., Dehghani, M. H., Heidari, M., Javedan, G., Ali, I., & Sillanpää, M. (2020). Methods for Preparation and Activation of Activated Carbon: A Review. *Environmental Chemistry Letters*, *18*(2), 393–415.

Hidayu, A. R., & Muda, N. (2016). Preparation and Characterization of Impregnated Activated Carbon from Palm Kernel Shell and Coconut Shell for CO2 Capture. *Procedia Engineering*, *148*, 106–113.

Kra, D. O., Allou, N. B., Atheba, P., Drogui, P., & Trokourey, A. (2019). Preparation and Characterization of Activated Carbon Based on Wood (Acacia auriculeaformis, Côte d'Ivoire). *Journal of Encapsulation and Adsorption Sciences*, *09*(02), 63–82.

Lee, C. L., Chin, K. L., H'ng, P. S., Rashid, U., Maminski, M., & Khoo, P. S. (2021). Effect Of Pretreatment Conditions on The Chemical–Structural Characteristics Of Coconut and Palm Kernel Shell: A Potentially Valuable Precursor for Eco-Efficient Activated Carbon Production. *Environmental Technology & Innovation*, *21*, 101309.

Maulina, S., Handika, G., Irvan, & Iswanto, A. H. (2020). Quality Comparison of Activated Carbon Produced From Oil Palm Fronds by Chemical Activation Using Sodium Carbonate versus Sodium Chloride. *Journal of the Korean Wood Science and Technology*, *48*(4), 503–512.

Nicholas, A., Hussein, M., Zainal, Z., & Khadiran, T. (2018). Palm Kernel Shell Activated Carbon as an Inorganic Framework for Shape-Stabilized Phase Change Material. *Nanomaterials*, *8*(9), 1-14.

Okoroigwe, E. C., Ofomatah, A. C., Oparaku, N. F., & Unachukwu, G. O. (2013). Production And Evaluation of Activated Carbon from Palm Kernel Shells (PKS) for Economic and Environmental Sustainability. *International Journal of Physical Sciences*, *8*(19), 1036–1041.

Priyanto, A., Malik, F., Muhdarina, M., & Awaludin, A. (2021). Adsorption and Characterization of Activated Sugarcane Bagasse Using Natrium Hydroxide. *Indonesian Journal of Chemical Research.*, *8*(3), 202–209.

Rambabu, N., Rao, B. V. S. K., Surisetty, V. R., Das, U., & Dalai, A. K. (2015). Production, Characterization, and Evaluation of Activated Carbons from De-Oiled Canola Meal for Environmental Applications. *Industrial Crops and Products*, *65*, 572–581.

Rashidi, N. A., & Yusup, S. (2021). Co-Valorization of Delayed Petroleum Coke-Palm Kernel Shell for Activated Carbon Production. *Journal of Hazardous Materials*, *403*, 123876.

Rodrfguez-Reinoso, F., & Sepúlveda-Escribano, A. (2001). Porous Carbons in Adsorption and Catalysis. *Handbook of Surfaces and Interfaces of Materials*, *5*, 309–335.

Schleicher, T., Hilbert, I., Manhart, A., & Hennenberg, D. K. (2019). Production of Palm Oil in Indonesia. *Country-Focused Commodity Analysis in the Context of the Bio-Macht Project. Freiburg/Bandung.*

Sekewael, S. J., Latupeirissa, J., & Johannes, R. (2015). Adsorption of Cd Metal Using Active Carbon from Cacao Shell (Theobroma cacao). *Indonesian Journal of Chemical Research*, *2(2)*, 204–197.

Tan, Y. Y., Bello, M. M., & Abdul Raman, A. A. (2021). Towards Cleaner Production in Palm Oil Industry: Advanced Treatment of Biologically-Treated POME Using Palm Kernel Shell-Based Adsorbent. *Cleaner Engineering and Technology*, *2*(100079), 1–9.

Tanasale, M. F. J. D. P., Latupeirissa, J., & Letelay, R. (2014). Adsorption of Tartrazine Dye by Active Carbon from Mahagony (swietenia Mahagoni Jacq) Rind. *Indonesian Journal of Chemical Research*, *1(2)*, 104–109.

Ulfah, M., Raharjo, S., Hastuti, P., & Darmadji, P. (2017). Adsorption of β-Carotene in Isopropyl Alcohol with Decolorized Activated Carbon as Model for β-Carotene Adsorption in Crude Palm Oil. *Indonesian Journal of Chemistry*, *17*(1), 105.

Zakaria, R., Jamalluddin, N. A., & Abu Bakar, M. Z. (2021). Effect of Impregnation Ratio and Activation Temperature on The Yield And Adsorption Performance of Mangrove Based Activated Carbon for Methylene Blue Removal. *Results in Materials*, *10*, 100183.