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Submission date: 15-Mar-2022 10:02PM (UTC+0700)

Submission ID: 1784906625

File name: 4._541-1602-2-CE.pdf (347.51K)

Word count: 4155

Character count: 21551



Activation of $ZnCl_2$ and KOH Carbon from Bark of Salak Wedi as a Material for Making Supercapacitor Electrodes

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Article Info

Article History

Received: 22 February 2022

Revised: 28 February 2022

Published: 25 March 2022

Keywords

Salak wedi, $ZnCl_2$, KOH, Elektrodes

Abstract

Activation of $ZnCl_2$ and KOH Carbon from bark of salak wedi as a material for making supercapacitor electrodes. An activated carbon is a potential material that can be used as electrodes in super capacitors. It has a higher energy density than batteries and fuel cells, and a higher power density than conventional capacitors. This research was carried out using activated carbon powder from the bark of salak wedi which was physically activated at a temperature of $500^\circ C$. The activation stage is chemical activation using $ZnCl_2$ and KOH activators, after which gradual activation is carried out, namely: carbon $ZnCl_2 + KOH (s)$ and carbon $KOH + ZnCl_2 (s)$. So that with the gradual activation, 4 variations of the sample will be obtained, namely: $ZnCl_2, KOH, carbon ZnCl_2 + KOH (s)$ and $carbon KOH + ZnCl_2 (s)$. The physical characteristics of the activated carbon from the bark of salak wedi were analyzed using a Scanning Electron Microscope (SEM) to see its powder morphology. While, X-ray diffraction (XRD) is used to examine the structure of the bark of activated carbon that has the potential to make electrodes of super capacitors. The results of SEM with graded activation had fewer pores, were not evenly distributed and tended to agglomerate compared to the activated carbon sample of salak bark which was activated once. This research has not yet reached the stage of making electrodes, it is hoped that in future research it can be applied to electrodes so that data such as capacity, voltage, current and others are obtained.

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Citations: Ardianti, A. D & Yuwita, P. E. (2022). Activation of $ZnCl_2$ and KOH Carbon from Bark of Salak Wedi as a Material for Making Supercapacitor Electrodes. Science Education and Application Journal, 4(1), 37-45.

INTRODUCTION

Supercapacitors are electrical energy storage devices that have some characteristics such as a longer life time compared to the batteries as well as simple principles and models, short charging times, high power density, safe because they do not contain corrosive materials and less poisonous materials (Fitriana, 2014). Super capacitors can provide at least 1000 times more energy than dielectric capacitors and 10 times more power than batteries. Super capacitors have a long life cycle of more than 500000 cycles (Zhou et al., 2015). Activated carbon is one of the kind of materials that has been widely used because it has a high surface area, chemical resistance, good electrical conductivity and an affordable price (Mardwianta, 2017). Activated carbon is a material that contains a large amount of free

carbon, where the free carbon has a high absorption capacity and has pores that increase its absorption due to its reactions with chemicals before or after carbonization (Waluyo et al., 2017).

Many studies on activated carbon have been carried out before this study. A preliminary study on the effect of temperature and concentration on carbon activation process from Halaban wood using ZnCl₂ and KOH has been carried out by Amanah with the results of the absorption efficiency of the two largest activators being ZnCl₂ with a value of 95.1% and KOH of 93.3% (Permata et al., 2019). The manufacture of activated carbon from coconut shells to reduce ammonia levels with KOH, NaCl and HCl activators was carried out by Nisa Nurhidayanti (Nurhidayanti, 2020). Research on variations in holding time of activated carbon activation temperature from kluwak shell as an electrode on a super capacitor with results that the longer holding time, the smaller the pore size, the larger the surface area and the greater the capacitance (Habibah, 2016). The analysis on the differences of the activator materials in the manufacture of super capacitor electrodes from coconut shell charcoal get the results of increasing the value of specific capacitance with chemical activation using KOH activator (Susanti et al., n.d.-a; Taer et al., 2018).

The factor that affects the amount of absorption of an activated carbon is the surface area. Various methods can be used to increase the surface area of activated carbon (Kearns et al., 2014). One of the ways is the use of activators. In addition, the structure of activated carbon also determines the surface area of activated carbon (Sayğl & Güzel, 2016). The structure of activated carbon is basically amorphous. With activation treatment on carbon can also increase the crystallinity of activated carbon (Ghosh et al., 2019). The crystallinity of activated carbon affects the surface area. With regularity on the crystal structure and Nano size makes a wider surface area (Jain et al., 2018).

Based on the references of previous studies, the researcher wanted to conduct research with several activation treatments on salak bark carbon and determine the morphology and structure of activated carbon which has the potential to be used as electrodes in super capacitors. Salak (*Salacca edulis* Reinw) comes from Southeast Asia (Mazumdar et al., 2019). This fruit has a shape resembling an egg. The skin of the fruit is brown and looks like snakeskin. Salak contains three pieces of seeds covered with white flesh. In Indonesia, there are many cultivars of salak, but most of them have an astringent taste. The skin of the fruit is covered with scales, the appearance looks like reptile skin. The edible part is the white flesh which is aromatic and translucent, tastes like a mixture of pineapple and banana. Each fruit contains 1 to 3 dark brown seeds. The flesh of the fruit is edible and consists of three lobes (Deininger et al., 2002). After peeling, the skin becomes waste and it is used as raw material for the manufacture of activated carbon in this study.

Activated carbon is an amorphous carbon from flat plates composed of C atoms covalently bonded in a flat hexagonal lattice with one C atom at each corner and has a hollow surface and a layered structure. High quality activated carbon is characterized by high surface area and pore volume (Hartanto & Ratnawati, 2010). The process of making activated carbon is carried out in two stages. The first stage is the carbonization process of raw materials which is carried out to produce charcoal and the second stage is an activation process to remove the hydrocarbons that coat the surface of the charcoal so that the porosity increases (Lempang et al., 2011).

The factor that affects the amount of absorption of an activated carbon is the surface area. Various methods can be used to increase the surface area of activated carbon. One way that is often used is the use of activators. In the activator research in stages, it produces a higher capacitance value. This capacitive property is related to a higher carbon surface area, whereas in carbon that only one activation is carried out it results in a lower capacitive

property (Jin et al., 2013). Chemical activation is generally done by conditioning the base material with a strong dehydrating agent, such as phosphoric acid or other chemicals such as HNO₃, ZnCl₂, CaCl₂ (Masriatini, 2018). According to its use, activated charcoal is classified as a chemical product and not an energy source such as charcoal or charcoal briquettes. Advanced charcoal processing technology into activated charcoal will provide great added value in terms of use and economic value (Hendra, 2007). The activation process is an important thing to note in addition to the raw materials used. Activation is a treatment of charcoal that aims to enlarge the pores, namely by breaking the hydrocarbon bonds or oxidizing the surface molecules so that the charcoal undergoes changes in properties, both physical and chemical, namely the surface area increases and affects the adsorption power (Ajayi et al., 2009). The activators used in this study were ZnCl₂ and KOH. The research is analysis of the structure of salak wedi activated carbon as a material for making supercapacitor electrodes. The activation stage is chemical activation using ZnCl₂ and KOH activators, after which gradual activation is carried out, namely: carbon ZnCl₂ + KOH (s) and carbon KOH + ZnCl₂ (s). So that with the gradual activation, 4 variations of the sample will be obtained, namely: ZnCl₂, KOH, carbon ZnCl₂ + KOH (s) and carbon KOH + ZnCl₂ (s) (Susanti et al., n.d.-a)

METHODS

Method and Procedure

Salak bark is carbonated in a furnace which is flowed with inert gas at a temperature of 500°C for approximately 1 hour which aims to omit volatile substances in the bark. Then the bark char-coal is rested to cool in a desiccator. After that, the bark charcoal produced was ground and sieved through a 100 mesh sieve and retained on a 200 mesh sieve to produce carbon powder (Bagheri & Abedi, 2009). The resulting carbon powder is then mixed with solid activator with a mass ratio of carbon powder's mass and activator, 1:4. The solid activator was dissolved with distilled water to a concentration of 20%. The carbon powder was then mixed into the solution and shaken for 20 hours with a magnetic stirrer. The activated carbon powder was then oven-dried then washed with distilled water and dilute HCl solution until the pH of the washing water reached 6-7 (Bagheri & Abedi, 2009). The washed carbon powder is then dried again in the oven. The activation stage is chemical activation using ZnCl₂ and KOH activators, after which gradual activation is carried out, namely: carbon ZnCl₂ + KOH (s) and carbon KOH + ZnCl₂ (s). So that with the gradual activation, 4 variations of the sample will be obtained, namely: ZnCl₂, KOH, carbon ZnCl₂ + KOH (s) and carbon KOH + ZnCl₂ (s) (Susanti et al., n.d.-a). The scanning electron microscope characterization was carried out to determine the surface morphology of the bark's carbon the identification of the pore distribution. The scanning electron microscope was tested with four magnifications, those are 500x, 1000x, 5000x and 10.000x (Kiswandono, 2014; Susanti et al., n.d.-b). As well as testing using XRD to see the structure of bark's activated carbon which has the potential to be used as electrodes in super capacitors.

RESULTS AND DISCUSSION

The formation of pores on the activated carbon of salak bark is shown by the surface morphology of the activated carbon from the Scanning Electron Microscope (SEM) which is shown in Figure 4 with a magnification of 2500 times.

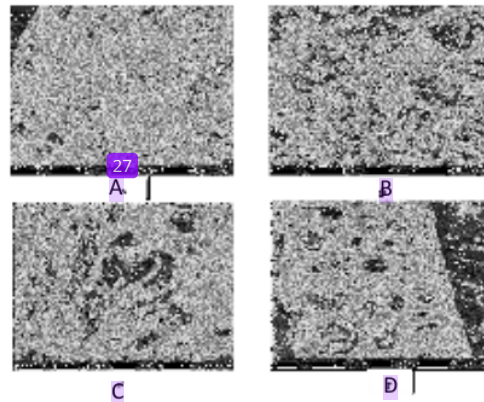


Figure 1. SEM results. (a) bark carbon with KOH activation (b) bark carbon with ZnCl₂ activation (c) bark carbon with graded activator Carbon ZnCl₂ + KOH (s) (d) bark carbon with graded activator Carbon KOH + ZnCl₂ (s)

In Figure 1(a) the carbon sample of salak bark with KOH activation shows that a large number of evenly distributed pores have been formed on the surface of the sample. In Figure 4(b) the carbon sample of salak bark with ZnCl₂ activation shows that pores are formed but the surface tends to be insulated and the number of closed pores is more -compared to the KOH activator. This is because the use of ZnCl₂ activator can form a ZnO phase in it. The SEM results of the ZnCl₂ + KOH(s) gradual activator are shown in Figure 4(c). The surface morphology of the sample shows the formation of pores but the number of pores is less and the surface is insulated. Meanwhile, Figure 4(d) shows the SEM results from the surface of the activated carbon of the salak bark with the gradual activator Carbon KOH + ZnCl₂ (s). The surface morphology of the sample shows the formation of pores but it is not evenly distributed and has the least number of pores compared to other activator variations. The SEM results show the pore size in the nanoscale. SEM results with gradual activation had fewer pores, were not evenly distributed and tended to agglomerate compared to the activated carbon sample of salak bark which was activated once. In addition, during the carbonization process, nitrogen gas is not flowed, it also affects the uneven distribution of pores on the surface of the activated carbon of the bark (Nurdiansah & Susanti, 2013).

The XRD diffraction pattern of a sample of salak bark carbon powder which was activated once with KOH and ZnCl₂ is shown in Figure 1.

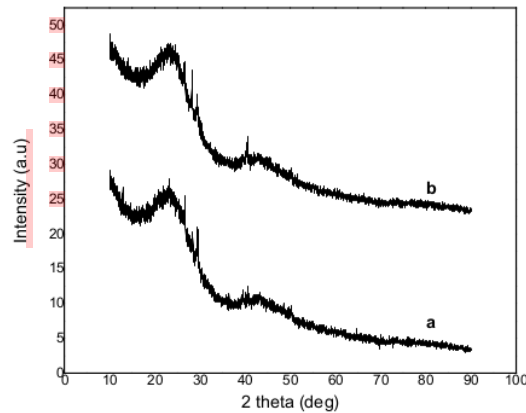


Figure 2 (a) shows the XRD pattern of samples of bark carbon powder activated with KOH, while Figure 1 (b) shows the XRD pattern of samples of bark carbon powder activated with ZnCl₂.

Figure 2(a) shows the XRD pattern of salak bark carbon samples with gradual activator of Carbon ZnCl₂ + KOH(s), while in Figure 2(b) shows XRD pattern of salak bark carbon samples which activated gradually with KOH + ZnCl₂(s). From all XRD diffraction results of salak bark carbon powder, an amorphous structure with broad peaks was formed. However, in samples of salak bark carbon powder which was activated once using ZnCl₂ activator in Figure 1(a) and salak bark carbon powder which was activated once using KOH activator in Figure 1(b), the diffraction peaks were higher. The increase in the diffraction peak indicates that the degree of crystallinity of the sample of salak bark carbon powder is also getting higher.

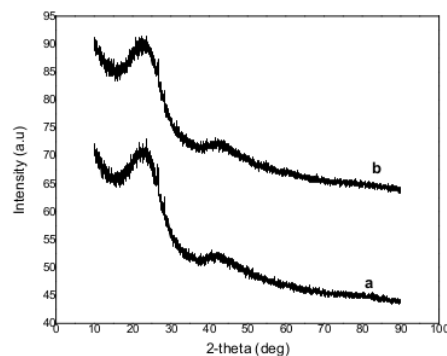
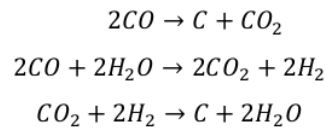


Figure 3. XRD results. (a) bark carbon with graded activator Carbon ZnCl₂ + KOH(s) (b) bark carbon with graded activator Carbon KOH + ZnCl₂(s)

The increase in the degree of crystallinity in the sample of salak bark carbon powder is similar to that of graphite, which is the main element of activated carbon. According to Kwieciriska, B, et al (2003) the possible reactions in the formation of graphite are as follows:



XRD pattern identification results to determine the formed phase using Match! Software is shown in Figure 3.

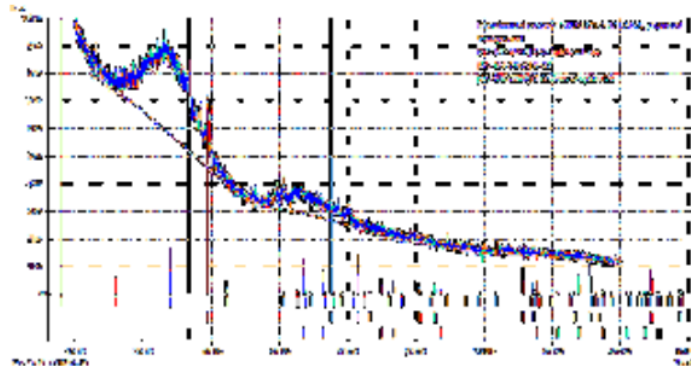


Figure 4. Identification results using the Match! software

In the X-ray diffraction pattern on samples of salak bark carbon powder which was activated once using KOH activator in Figure 1(a) and salak bark carbon powder which was activated once using ZnCl₂ activator in Figure 1(b) showed an amorphous structure at the highest peak at angle at of 26.50°-26.70° indicates the formation of a 2H Carbon Graphite phase with secondary data PDF 96-110-0004. In addition, the sample XRD pattern shows the formation of the Carbon Dioxide phase with secondary data PDF 96-151-7803, which is indicated by a peak at an angle of 28.38°-29.48°. In the XRD pattern, Graphite Nitrate phase is also formed with secondary data PDF 96-100-0066 which shows the highest peak at an angle of 39.56° and 40.49°. Carbon Graphite 2H phase formed has a crystal system and a trigonal R-3m space group. While the Carbon Dioxide phase has a crystal system and space group I-4 2 d tetragonal and Graphite Nitrate which is formed from activated bark carbon powder has a crystal system and a trigonal R-3m space group. Figure 2(a) shows the XRD pattern of the salak bark carbon sample which is activated in stages, which is activated with ZnCl₂ + KOH(s) and in Figure 2(b) shows the XRD pattern of the salak bark carbon sample which is activated in stages, which is activated with KOH(s) + ZnCl₂ shows an X-ray diffraction pattern, formed an amorphous structure and has a lower diffraction peak. It can be seen that the highest peak is formed from the diminishing X-ray diffraction. The diffraction peak at an angle of 26.50°-26.70° indicates the formation of a 2H Carbon Graphite phase with secondary data PDF 96-110-0004 and an angle of 28.38°-29.48° indicates the formation of a Carbon Dioxide phase with secondary data PDF 96-151-7803. The more gentle the diffraction pattern formed, indicates that the sample has a more amorphous structure.

CONCLUSION

The carbon sample of salak bark with KOH activation shows that a large number of evenly distributed pores have been formed on the surface of the sample. The surface

morphology of the sample shows the formation of pores but the number of pores is less and the surface is insulated. Diffraction results of salak bark carbon powder, an amorphous structure with broad peaks was formed. In addition, the sample XRD pattern shows the formation of the Carbon Dioxide phase with secondary data PDF 96-151-7803, which is indicated by a peak at an angle of 28.38°-29.48°. In the XRD pattern, Graphite Nitrate phase is also formed with secondary data PDF 96-100-0066 which shows the highest peak at an angle of 39.56° and 40.49°. From the carbon structure of the bark formed, it has indicated the formation of activated carbon which is marked by the formation of a graphite structure which is a characteristic of the structure of activated carbon (Rodríguez Correa et al., 2017). Morphology of salak bark carbon samples with KOH activation showed that a large number of uniform pores had been formed on the sample surface. In bark carbon with ZnCl₂ activation, it shows that pores are formed but the surface tends to be insulated and the number of closed pores is more. The surface morphology of the sample from the stratified activator Carbon ZnCl₂ + KOH(s) shows the formation of pores but the number of pores is smaller and the surface is insulated. While the SEM results from the surface of the activated carbon of the bark of bark with activator graded Carbon KOH + ZnCl₂ (s) it appears that pores are formed but are not evenly distributed and have the least number of pores compared to other activators variations. The results of SEM with graded activation had fewer pores, were not evenly distributed and tended to agglomerate compared to the activated carbon sample of salak bark which was activated once.

SUGGESTION

This research has not yet reached the stage of making electrodes, it is hoped that in future research it can be applied to electrodes so that data such as capacity, voltage, current and others are obtained.

ACKNOWLEDGMENTS

We thank the Ministry of Research, Technology and Higher Education DRPM for funding this research. This research is included in the Beginner Lecturer Research Grant scheme for the 2021 Fiscal Year.

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