Characterization of Salak Wedi Activated Carbon Structure Using Activator Materials as Materials for Making Supercapacitor Electrodes

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Abstract

An activated carbon is a potential material that can be used as electrodes in super capacitors because it has a higher energy density than batteries and fuel cells and a higher power density than conventional capacitors. The previous studies have been making activated carbon from coconut shells with ZnCl2 activator to produce activated carbon with large specific surface area results in the manufacture of super capacitor electrodes. While this research was carried out using activated carbon from the bark of *salak wedi* (Snake fruit) with a variety of gradual activators ZnCl2 and KOH. 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 tests using X-ray diffraction (XRD) is used to see the structure of the bark of activated carbon that has the potential to be used as electrodes in super capacitors.

Keywords : Supercapasitors, activated carbon, Salak, Activation of $ZnCl_2$ and KOH

1. Background

Super capacitors are electrical energy storage devices that have some characteristics such as a longer life time (when they are compared to batteries), simple principles and models, short charging times, high power density, safe because they do not contain corrosive materials and less poisonous materials [1]. Super capacitors can provide at least 1000 times more energy than dielectric capacitors and 10 times more power than batteries and super capacitors have a long life cycle of more than 500000 cycles [2]. 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 [3]. 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 [4].

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% [5]. The manufacture of activated carbon from coconut shells to reduce ammonia levels with KOH, Nacl and HCl activators was carried out by Nisa Nurhidayanti[6]. 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 [7]. 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 [8]

Salak (Salacca edulis Reinw) comes from Southeast Asia. This fruit has a shape resembling an egg. The skin of the fruit is brown and looks like snake skin. *Salak* contains three pieces of seeds covered with white flesh. In Indonesia there are many cultivars of *salak*, but most of them have astringent taste. The skin of the fruit is covered with regular scales, giving it the appearance of a 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 [9]. After peeled, the skin becomes waste and in this study is used as raw material for the manufacture of activated carbon.

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 of the ways is the use of activators. In addition, the structure of activated carbon also determines the surface area of activated carbon. The structure of activated carbon is basically amorphous. With activation treatment on carbon can also increase the crystallinity of activated carbon. The crystallinity of activated carbon affects the surface area. With regularity on the crystal structure and Nano size makes a wider surface area.

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. The activators used in this study were $ZnCl_2$ and KOH. The research is "Structure Analysis on *Salak Wedi's* Activated Carbon Using Activator Materials as the Materials in Making Super Capacitor Electrodes".

2. Methods

a. Sample making procedure

The biomass used in this research is bark waste of *salak wedi*. The raw material of the *salak* bark is cleaned with water then dried in an oven at 110°C of the temperature to omit the water content due to the washing process.

b. Carbonation 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 charcoal 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. [10]

c. Activation Process of *Salak* Bark Activated Charcoal

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[10]. The washed carbon powder is then dried again in the oven. 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). [8]

d. Sample Test

After that, the samples obtained were tested to characterize the physical properties of the activated carbon using *scanning electron microscope*. *The scanning electron microscope* characterization was carried out to determine the surface morphology of the bark's carbon by looking at the pore distribution. *The scanning electron microscope* was tested with four magnifications, those are 500X, 1000X, 5000X and 10,000X. 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.

3. Results and Discussion

A. XRD test results

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.

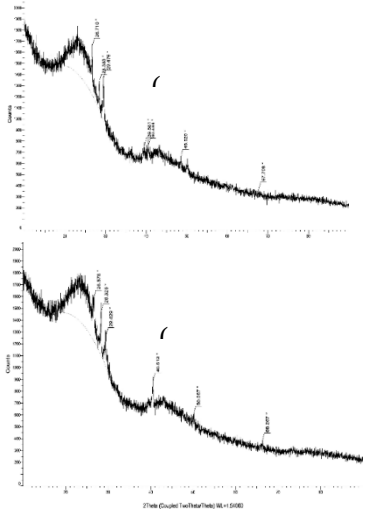

Figure 1. XRD results (a) bark carbon with KOH activation (b) bark carbon with ZnCl2 activation

Figure 1 (a) shows the XRD pattern of samples of bark carbon powder activated with ZnCl2, while Figure 1 (b) shows the XRD pattern of samples of bark carbon powder activated with KOH. XRD diffraction pattern sample of bark carbon powder which gradually activated by activated carbon ZnCl2 + KOH (s) and carbon KOH + ZnCl2 (s) is shown in Figure 2.

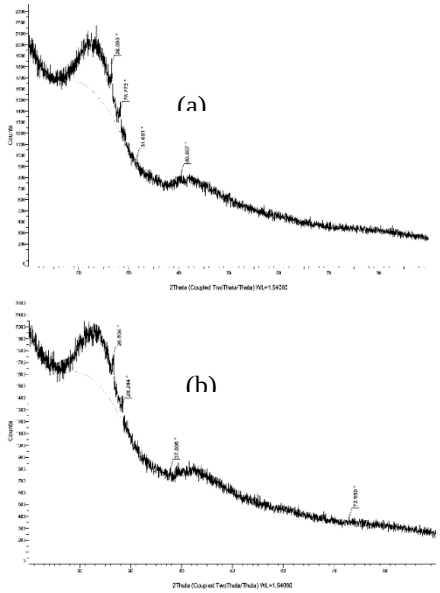

Figure 2. XRD results. (a) bark carbon with graded activator Carbon ZnCl2 + KOH(s) (b) bark carbon with graded activator Carbon KOH + ZnCl2(s)

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. 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:

$$2CO \longrightarrow C+CO_2$$

$$2CO + 2H_2O \longrightarrow 2CO_2+2H_2$$

$$CO2 +2H_2 \longrightarrow C+ 2H_2O$$

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

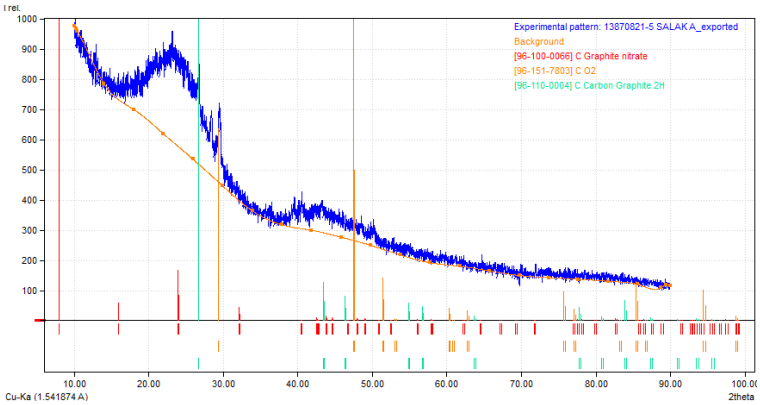

Figure 3. Identification results using the Match! software

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 26.50o-26.70o 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 28.38o-29.48o. 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 39.56o and 40.49o. *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 26.50o-26.70o indicates the formation of a *2H Carbon Graphite* phase with secondary data PDF 96-110-0004 and an angle of 28.38o-29.48o 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. However, 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.

B. SEM Test Result

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.

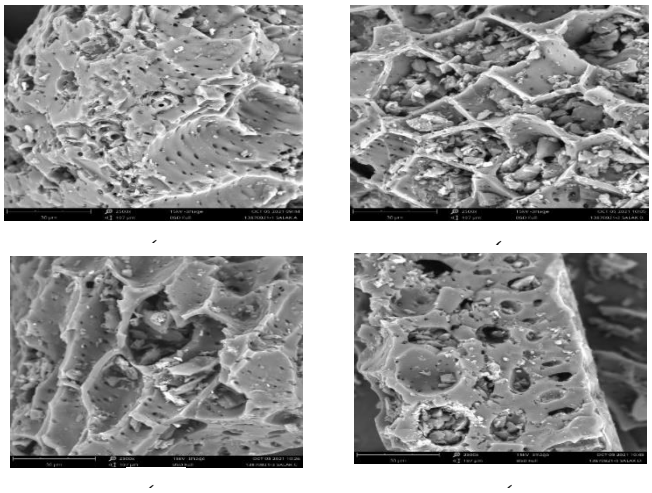

Figure 4. SEM results. (a) bark carbon with KOH activation (b) bark carbon with ZnCl2 activation (c) bark carbon with graded activator Carbon ZnCl2 + KOH (s) (d) bark carbon with graded activator Carbon KOH + ZnCl2 (s)

In Figure 4(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. [11]

4. Conclusion

In the X-ray diffraction pattern on samples of *salak* bark carbon powder which was activated once using KOH activator and using ZnCl2 activator showed an amorphous structure at the highest peak at an angle of 26.50o-26.70o, indicating the formation of *Carbon Graphite 2H* 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 28.38o-29.48o. In the XRD pattern, the *Graphite Nitrate* phase is also formed with secondary data PDF 96-100-0066 which shows the highest peak at an angle of 39.56o and 40.49o. In the XRD pattern of *salak* bark carbon samples that were activated in stages, which activated with ZnCl2 + KOH (s) and activated with KOH (s) + ZnCl2, the X-ray diffraction pattern formed an amorphous structure and had a lower diffraction peak with a diffraction peak at an angle of 26,50o-26,70o, indicates the formation of *Carbon Graphite 2H* phase with secondary data PDF 96-110-0004 and angle 28,38o-29,48o indicates the formation of *Carbon Dioxide* phase with secondary data PDF 96-151-7803. The morphology of *salak* bark carbon samples with KOH activation showed that a large number of evenly distributed 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 more number of closed pores. 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 *salak*

bark with gradual activator Carbon KOH + ZnCl2 (s), appears that pores are formed but not evenly distributed and have the least number of pores compared to other activator variations. 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.

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