



# Manufacture of Supercapacitor Electrode from Durian Shell Waste Using Pyrolysis and Chemical Activation Method with Activator Variation

Tantra Diwa Larasati<sup>1\*</sup>, Ari Susandy Sanjaya<sup>1</sup>, Melvin Felixander Yovandra<sup>1</sup>  
and Muhammad Reyhandi Rizki Fahrezi<sup>1</sup>

<sup>1</sup> Mulawarman University, Samarinda East Kalimantan 75119, Indonesia  
tantralarasati@ft.unmul.ac.id

**Abstract.** A supercapacitor is a device used to store energy through electrochemical processes. Supercapacitors are used in many applications, such as batteries for electric cars. The material used for the electrodes determines the performance of the supercapacitor. Activated carbon is one of the most commonly used electrode materials. Biomass with a high lignin and cellulose content, such as durian shells, produces activated carbon. This research aims to evaluate the potential of durian shells as an electrode material for supercapacitors. The method used is to form carbon from durian shells through pyrolysis and use chemicals to make activated carbon. Pyrolysis is carried out at several temperature variations to determine the ideal temperature for carbonization—carbon activation using several chemicals to determine the best activator. The activated carbon is applied for the Proximate test, BET, and SEM characterization. Proximate test results indicate that the carbon generated by pyrolysis at 400°C has the highest fixed carbon content. The activation process with the ZnCl<sub>2</sub> solution showed the highest absorption rate. SEM characterization showed that activated carbon pyrolyzed at 300°C and activated with ZnCl<sub>2</sub> had a more porous surface morphology with different pore sizes. BET characterization using the same activated carbon shows the carbon's specific surface area is 629 m<sup>2</sup>/gram. The activated carbon from the durian shell has enough capacitance potential to be used as an electrode material.

**Keywords:** Durian, Pyrolysis, Carbon Active, BET, SEM.

## 1 Introduction

Durian is one of the fruits widely consumed by Indonesians. Badan Pusat Statistik Indonesia noted that durian production in Indonesia reached 1.35 million tons in 2021. The high production value also causes the waste produced, in this case, durian shells, to increase. Most of this biomass waste will be disposed of, and no further treatment will be provided.

Durian is one of the fruits with a relatively high cellulose content, around 50-60%, and a lignin content of 5%. Typically, a high carbon content coexists with a high cellulose content. Most of these ingredients are found in durian shells. This fruit's shell extends to almost a quarter of the fruit, allowing for the processing of many components of biomass waste into various materials, including activated carbon [1].

There have been several attempts to make use of this durian waste as such as food wrappers (edible film) [2], bio-pesticides [3], bio-briquettes [4], bio-batteries [5], and activated carbon [6-8].

Activated carbon can be produced using raw materials from biomass waste from durian shells. A carbon compound with a large surface area and adsorption power is called activated carbon. The processes of carbonization and activation are typically used to produce activated carbon. Carbonation is the imperfect combustion process of pyrolysis (limited air) of carbon-containing materials [9]. The combustion and removal of other organic compounds during this process will cause biomass composition, such as cellulose and lignin, to break down into carbon solids.

Pyrolysis is a technology for the chemical decomposition of organic matter through a heating process with no or little oxygen or other reagents. Pyrolysis occurs in an atmospheric reduction reactor (vacuum) between 250°C and 800°C [10]. The final product of the pyrolysis process will produce three compounds, namely gas, solid and liquid, in the form of carbon solids, liquids in the form of tar mixtures (bio-oil) and several other substances. In general, the pyrolysis process runs for 4-7 hours with a temperature above 300°C, but this condition is influenced by the physical properties of the raw materials [11].

Charcoal is treated with activation, which aims to increase the size of its pores by oxidizing surface molecules or breaking hydrocarbon bonds. This process causes charcoal to change chemically and physically, increasing its volume, surface area, and absorption capacity [12]. The activation stage is the process of removing impurities and opening closed carbon pores. Activation can be done physically by heating or chemically using an activator such as  $H_2SO_4$  [13]. Activated carbon increases the surface area and pores in carbon by breaking the carbon chain and breaking down organic compounds in carbon. This condition will have an impact on the quality of activated carbon.

There are several methods to determine the outcome of carbon activation, including Scanning Electron Microscopy (SEM) observations, Brunauer-Emmett-Teller (BET) tests, and Proximate tests. The Proximate test was carried out to determine several contents such as moisture, ash, volatile matter, and carbon content in a sample. The SEM test provides detailed surface information by tracking samples in a raster pattern with electron beams [14]. The BET test was carried out using the SAA (Surface Area Analyzer) tool to determine the diameter and volume of the pore, as well as the specific surface area of the material [15].

For example, activated carbon can be used in supercapacitor cells [1]. Supercapacitors are one of the most efficient energy storage systems, and they can store energy through electrical double-layering and faric reactions. The material's active surface area is used by an electrical double-layer capacitor (EDLC), which stores electric charge and performs incredibly well over an extended length of time. One method to enhance EDLC's supercapacitor performance is to create carbon with a large surface area [16]. There have been numerous studies on producing supercapacitor cell electrodes from durian shell, where the highest specific capacitance supercapacitor cells produced reaching 89.05 F/g [1].

This research was conducted to determine the potential use of durian shells as activated carbon as a supercapacitor material. Durian shells are biomass waste that is pyrolyzed into carbon and activated with activators to form activated carbon. The results

are tested to determine the carbon content and quality of the activated carbon produced and whether it is suitable for use as a supercapacitor.

## **2 Materials and Methods**

### **2.1 Dehydration**

Dehydration is the process of drying the water contained in the raw materials. Dehydration is a process of removing water contained in biomass by drying in the sun and drying ovens. The raw materials are cleaned of impurities and cut into 2 cm long pieces at this stage. Then, the raw materials are heated in the oven at a temperature of 120°C for 24 hours until they dry or lose their moisture content.

### **2.2 Carbonization**

Carbonization in the production of activated carbon usually occurs through the pyrolysis process. The dried durian shells are burned in a pyrolysis reactor at combustion temperatures of 250°C, 300°C, 350°C, and 400°C. This pyrolysis process uses charcoal and coconut fiber, with the pyrolysis time corresponding to the burning time of the fuel. The resulting carbon is grounded in the porcelain until it turns into powder.

### **2.3 Activation**

The carbon produced by the carbonization process is immersed in various activator solutions, such as  $ZnCl_2$  and  $H_2SO_4$  solutions, with an activator concentration of 1 M for 24 hours. The activated carbon is then washed with an aqueous solution until neutral. The washed activated carbon is dried in an oven at 150°C for 24 hours.

### **2.4 Characterization**

Activated carbon is characterized to determine the suitability/feasibility of activated carbon as a raw material for supercapacitor electrodes. The analysis is an elemental analysis to determine the activated carbon content. Proximate test to determine the composition of carbon and ash in activated carbon, Scanning Electron Microscopy (SEM) to identify the morphological structure of activated carbon and Brunauer-Emmett-Teller (BET) to determine the specific surface area of activated carbon when adsorbing gases.

## **3 Discussion**

### **3.1 Analysis of Pyrolysis Results**

Durian shell has a relatively high moisture content [17]. Therefore, durian must go through a drying process to reduce the moisture content in the durian skin. The water

content in the biomass, in this case, the durian shell, needs to be reduced to facilitate the pyrolysis process. Dry biomass accelerates the process of temperature increase and mass decrease.

**Table 1.** Durian Shell Moisture and Pyrolysis Testing Results.

| Parameter                    | Temperature |             |            |            |
|------------------------------|-------------|-------------|------------|------------|
|                              | 250°C       | 300°C       | 350°C      | 400°C      |
| <b>Mass Before Drying</b>    | 330 gram    |             |            |            |
| <b>Mass After Drying</b>     | 62.8 gram   |             |            |            |
| <b>Moisture Content</b>      | 80.97%      |             |            |            |
| <b>Mass Before Pyrolysis</b> | 119.90 gram | 115.70 gram | 94.56 gram | 98.98 gram |
| <b>Mass After Pyrolysis</b>  | 38.50 gram  | 47 gram     | 45.90 gram | 50.50 gram |
| <b>Carbon Yield</b>          | 32.11%      | 40.62%      | 48.54%     | 51.02%     |

The results of the moisture content calculation show that the durian shell has a relatively high moisture content. The amount of water contained in biomass or charcoal can affect the pyrolysis process as it reduces the effectiveness of the evaporation of water vapor and carbonization processes [18]. This factor also affects the time and stability of the pyrolysis process, and the amount of ash and carbon produced. The lower the ash content of biomass, the more carbon can be made [19].

The dried durian shells were pyrolyzed at 250°C, 300°C, 350°C, and 400°C with a total pyrolysis time of 74.55 minutes, 77.46 minutes, 83.49 minutes, and 98.67 minutes. The duration of the pyrolysis process depends on several factors, such as the pyrolysis temperature, the combustion time, the fuel type, and the environmental conditions at the time of the process. The results of pyrolysis at 400°C showed the highest yield percentage. The pyrolysis process at too low a temperature results in some of the durian shells not being fully carbonized, while at too high a temperature, the amount of ash produced increases.

**Table 2.** Carbon Proximate Test Results.

| Parameter               | Temperature |        |        |        |
|-------------------------|-------------|--------|--------|--------|
|                         | 250°C       | 300°C  | 350°C  | 400°C  |
| <b>Moisture Content</b> | 2.12%       | 1.88%  | 1.43%  | 0.96%  |
| <b>Ash Content</b>      | 28.85%      | 27.35% | 30.56% | 31.32% |
| <b>Volatile Matter</b>  | 30.72%      | 30.15% | 20.82% | 16.59% |
| <b>Fixed Carbon</b>     | 38.31%      | 40.62% | 47.19% | 51.13% |

The carbon or charcoal formed is analyzed using the Proximate test. The results show that the higher the carbonization temperature, the higher the evaporated water content [20]. This condition happens because the higher the pyrolysis temperature, the more open the pores of the charcoal are, so that when there is direct contact between hygroscopic charcoal and air, the charcoal absorbs water vapor and causes an increase in moisture content [21].

The ash content produced shows that the higher the pyrolysis temperature, the higher the ash content produced [22]. Furthermore, this may be due to the high tar content and organic minerals covering the charcoal's pores [23]. The amount of ash formed can vary

depending on the type of biomass, the temperature and duration of the pyrolysis process, and the water content of the raw materials.

The volatile content of charcoal decreases as the carbonization temperature increases. This condition happens because, at high temperatures, the decomposition process of non-carbon compounds occurs ideally [24]. The high carbonization temperature causes many volatile compounds to evaporate and results in low volatility values.

The charcoal's moisture content, ash, and volatile substances influence the fixed carbon content. The lower the moisture content and ash, the higher the fixed carbon value. The fixed carbon content is the amount of pure carbon contained in charcoal. The higher temperature in the carbonization process dramatically influences the quality of charcoal, including the value of fixed carbon [21]. The fixed carbon value obtained from this pyrolysis process increases with an increase in pyrolysis temperature. The results of the Proximate test showed that the carbon produced by pyrolysis at 400°C has the highest fixed carbon content.

**Table 3.** Results of Activated Carbon Using ZnCl<sub>2</sub> Activator.

| Parameter                     | Temperature |           |           |           |
|-------------------------------|-------------|-----------|-----------|-----------|
|                               | 250°C       | 300°C     | 350°C     | 400°C     |
| <b>Mass Before Activation</b> | 3.00 gram   | 3.00 gram | 3.00 gram | 3.00 gram |
| <b>Mass After Activation</b>  | 8.41 gram   | 7.98 gram | 8.01 gram | 8.10 gram |
| <b>Mass After Drying</b>      | 3.06 gram   | 3.56 gram | 4.10 gram | 4.62 gram |
| <b>Mass Improvement</b>       | 101.98%     | 118.62%   | 136.64%   | 153.31%   |

**Table 4.** Results of Activated Carbon Using H<sub>2</sub>SO<sub>4</sub> Activator.

| Parameter                     | Temperature |           |           |           |
|-------------------------------|-------------|-----------|-----------|-----------|
|                               | 250°C       | 300°C     | 350°C     | 400°C     |
| <b>Mass Before Activation</b> | 3.00 gram   | 3.00 gram | 3.00 gram | 3.00 gram |
| <b>Mass After Activation</b>  | 6.29 gram   | 5.99 gram | 6.68 gram | 6.89 gram |
| <b>Mass After Drying</b>      | 3.02 gram   | 3.35 gram | 4.08 gram | 4.41 gram |
| <b>Mass Improvement</b>       | 100.78%     | 111.68%   | 135.98%   | 146.98%   |

The chemical activation of the activated carbon is carried out with a ZnCl<sub>2</sub> salt activator and H<sub>2</sub>SO<sub>4</sub> acid activator. The carbon activation process can be identified by the increase in mass of each sample based on the activator used. Carbon samples pyrolyzed at 400°C showed more significant mass gain than those at lower temperatures. This condition happens because the increase in temperature causes more carbon to be formed and the pores on the carbon surface to enlarge. This condition increases the amount of activator substances absorbed onto the carbon surface after drying.

The usage of ZnCl<sub>2</sub> as an activator showed the highest mass increase value. ZnCl<sub>2</sub> solution can dissolve minerals and organic compounds from pyrolyzed carbon by degrading compounds such as cellulose in them. ZnCl<sub>2</sub> activators hydrate and produce aromatic structures to form pores from the process and improve the structure of carbon pores [25]. Meanwhile, H<sub>2</sub>SO<sub>4</sub> increases the surface area by decomposing carbon bonds and dissolving organic compounds from carbon [26].

### 3.2 Material Characterization

SEM testing aims to determine the surface morphology of activated carbon and the average particle size. The morphological form of each substance is different depending on the presence of volatile compounds that are not entirely decomposed [24].

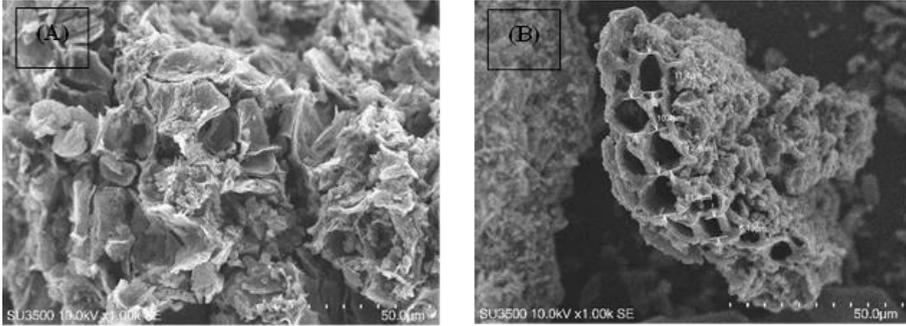


Fig. 1. SEM Results of (A) AC-H<sub>2</sub>SO<sub>4</sub> and (B) AC-ZnCl<sub>2</sub>

The activated carbon obtained indicates the formation of pores and cracks on the surface of the fibers. The pore structure obtained by the activation process with ZnCl<sub>2</sub> is not uniform with different pore sizes. This statement supported the results obtained by using various raw materials but using ZnCl<sub>2</sub> activators [27], [28]. Meanwhile, the activation process with H<sub>2</sub>SO<sub>4</sub> shows smoother pores that tend to be more evenly distributed [26]. However, activated carbon with the activator ZnCl<sub>2</sub> showed a better pore structure.

The high specific surface area of the adsorbent can produce considerable adsorption power. Activated carbon surface modification aims to create functional groups on the carbon surface by adding an oxygen structure, which causes the surface area to become more significant. This condition will increase the capacitance value of activated carbon as an electrode [29].

Table 5. BET Test Results.

| Activating Agent               | Temperature (°C) | Surface Area (m <sup>2</sup> /g) |
|--------------------------------|------------------|----------------------------------|
| H <sub>2</sub> SO <sub>4</sub> | 300              | 201                              |
|                                | 400              | 328                              |
| ZnCl <sub>2</sub>              | 300              | 629                              |
|                                | 400              | 406                              |

The BET surface area of AC-ZnCl<sub>2</sub> shows the highest BET surface area at a temperature of 300°C. This condition can happen because the heat treatment's temperature increase results in slight weight loss, structure organization, and shrinking of the carbon particles, considerably reducing the porosity surface area [25]. Increasing temperatures at lower temperatures causes more volatile compounds to be released, which helps open pores and increase surface area. However, at higher temperatures, above 400-500°C,

pore expansion occurs, which leads to the destruction of the pore structure formed [30].  $\text{ZnCl}_2$  as an activator can increase surface area and good pore structure, but a concentration of  $\text{ZnCl}_2$  that is too high can reduce the surface area [31].

## 4 Conclusions

Durian shell is one of the biomass wastes that can be used for various applications, including activated carbon for supercapacitors. Carbon from durian shells is produced by pyrolysis at an optimal temperature of 400°C with a yield of 51.02%. The carbon produced has a high fixed carbon value of 51.13%. The activation process with  $\text{ZnCl}_2$  showed the highest percentage increase of 153.31% compared to  $\text{H}_2\text{SO}_4$ . Characterization with SEM showed that activated carbon with  $\text{ZnCl}_2$  activator had different pore sizes and non-uniform pore structures. In contrast, the results of the BET test showed that the largest surface area of activated carbon with  $\text{ZnCl}_2$  activator at 300°C with a value of 629 m<sup>2</sup>/g.

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