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Etlingera elatior leaf agricultural waste as activated carbon monolith for supercapacitor electrodes

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Abstract. Recently, biomass waste has become the focus of several researchers because it has promising potential when processed into porous activated carbon. Abundant availability, uncomplicated processing, and more economical are the reasons for choosing biomass as the basic material for making carbon electrodes for electric energy storage supercapacitors. In this study, *Etlingera elatior* waste biomass is processed into activated carbon by heating at high temperature and impregnation of 0.5 M $ZnCl_2$. The monolith sample was optimized through a single-stage integrated high-temperature pyrolysis process. Where the process of carbonization of N_2 gas from a temperature of 30 °C to 600 °C followed by a physical activation process of CO_2 gas to a temperature of 800 °C. Determination of the physical properties of the electrodes through density characterization, while the electrochemical properties were analyzed by cyclic voltammetry and galvanostatic charge discharge methods. Cyclic voltammetry and galvanostatic charge discharge analysis were performed with 1 M Na_2SO_4 aqueous electrolyte at a voltage of 0–1 V and a scan rate of 1 mV/s. Furthermore, the high electrochemical behavior of the CV method was found to be 108 F/g, while for the gcd method, the specific capacitance was much higher at 148 F/g at a constant current density of 1.0 A/g. Further calculations found an energy density of 8.23 Wh/kg and a power density of 161 W/kg. These results support the optimization of 0.5 M $ZnCl_2$ impregnated *Etlingera elatior* leaves as the base material for activated carbon electrodes to increase the supercapacitor capacitance.

1. Introduction

Indonesia is the country with the 4th highest population in the world after China, India and the United States with a total population of 271,349,889 million people. The high rate of population growth, especially in Indonesia, has an impact on meeting the needs of life, especially basic needs in the food sector, which is an important concern. As a result, businesses in the agricultural and plantation sectors have increased. The increase in the agricultural sector not only has a positive impact, but there is an increase in the production of waste that is also generated [1]. Agricultural waste includes organic waste, such as corn cobs, bagasse, coconut husks, *Etlingera elatior* leaves and others.

Organic agricultural waste is usually used as a basic material for compost and for animal feed. However, not all waste can be treated traditionally so that a lot is still wasted and has the potential to pollute the environment, especially clean water and air. Organic waste has a large enough carbon content, which can be used as a basic material as porous activated carbon which increases the use value of biomass waste [2,3]. One of the applications of this biomass-based porous carbon is as a based material for energy storage devices such as supercapacitors [4,5]. The preparation of activated carbon from biomass materials for supercapacitor electrodes as an energy storage device is very promising because it provides high surface area, diverse natural pore structures, high conductivity and good thermal stability [6,7]. In addition, its abundant availability, more economical price and excellent physico-chemical properties are one of the reasons for choosing biomass for supercapacitor electrodes [8,9].

Conversion of biomass waste into activated carbon is performed by various processes including carbonization, chemical activation, physical activation or a combination of the three [10,11]. The process of carbonization and physical activation significantly affects the formation of pore structures especially their combination in the conversion of waste biomass into porous activated carbon [12]. Their process is performed by flowing certain gases such as N_2, Ar, CO_2, and H_2O into the furnace tube [13]. Furthermore, activated carbon for the electrical energy storage supercapacitor of the electrochemical double layer (EDLC) is strongly influenced by its high porosity contributing to the specific surface area [14,15]. Good ion absorption, energy production and power density are strongly influenced by the availability of pores such as the combination of existing micro and meso pores [16,17]. A good combination of micro and meso pores can increase the number of stored ions and increase the electrode surface area [18,19]. The development of pores is largely determined by the chemical activation process involving chemical activating agents such as KOH, NaOH, H_2SO_4, H_3PO_4, $ZnCl_2$, and others [20,21]. $ZnCl_2$ has a role in inhibiting the formation of ar and accelerating the evaporation process of volatile compounds during the carbonization process [22]. According to the International UNION of Pure and Applied Chemistry (IUPAC), the pore size of activated carbon is divided into small micropores from 2 nm, meso pores from 2 nm to 50 nm, and large macropores from 50 nm [23,24].

Etlingera elatior is a type of plant that can live in tropical climates, including a type of shrub with a green midrib. *Etlingera elatior* is used for spices, has a distinctive aroma and taste for food and is efficacious for medicine. *Etlingera elatior* flowers are separated from their leaves to be sold or processed into kitchen spices, while the leaves are left to be thrown away as waste. This *Etlingera elatior* leaf waste has a lignocellulosic component that has high potential as a source of porous activated carbon for supercapacitor electrodes.

In this study, *Etlingera elatior* leaf waste was converted into porous activated carbon as the electrode base material for the supercapacitor. The porous carbon is prepared by a chemical activation process of $ZnCl_2$ and pyrolysis (one-step integrated carbonization and physical activation). Furthermore, the porous carbons are retained in the monolith form by maximizing their self-adhesive properties. The material properties were evaluated by reducing the monolith dimensions of activated carbon including mass, thickness, diameter, volume and density. Moreover, the electrochemical properties of the supercapacitors were evaluated by means of cyclic voltammetry and galvanostatic charge discharge techniques. The 0.5 M $ZnCl_2$ activated sample confirmed the high capacitive properties of 108 F/g in the 1 M Na_2SO_4 aqueous electrolyte. These results clearly confirm the potential of *Etlingera elatior* leaf waste as a porous carbon originating material for supercapacitor electrodes.

2. Materials and methods

Etlingera elatior (EE) leaf of porous carbon as the based electrode material for supercapacitors were obtained from agricultural waste in Pekanbaru, Riau province. The *Etlingera elatior* waste was cut into pieces in the size range of 3–5 cm to facilitate the process of evaporation of the water content when dehydrated in the sunlight and continued to oven dried at a temperature of 110 °C. The sample was pre-carbonized for 2.5 hours using a vacuum oven at a temperature of 250 °C to evaporate the remaining water content, preventing dirt, further moisture and producing a biochar. Then, the sample was crushed manually using a mortar and ball milling tool which aims to smooth the carbon particle size thus it passes during the 60 µm sieving process. Next, the carbon powder was chemically activated with 0.5 M $ZnCl_2$ using a hot plate at a temperature of 80 °C. Activated carbon powder is molded into monolith pellets using a hydraulic press. Samples in the form of pellets with an average mass of 0.7 g were prepared as many as 20 pieces. After that, the carbon pellets are carbonized and the physical activation in one step integrated pyrolysis. Carbonization is performed from room temperature to a temperature of 600 °C in an N_2 gas environment followed by a physical activation process up to 800 °C in a CO_2 gas environment. Finally, the sample was washed using distilled water to neutralize its pH which was then polished in a thickness range of 0.2–0.3 mm. In detail the process of prepared activated carbon is shown in Figure 1.

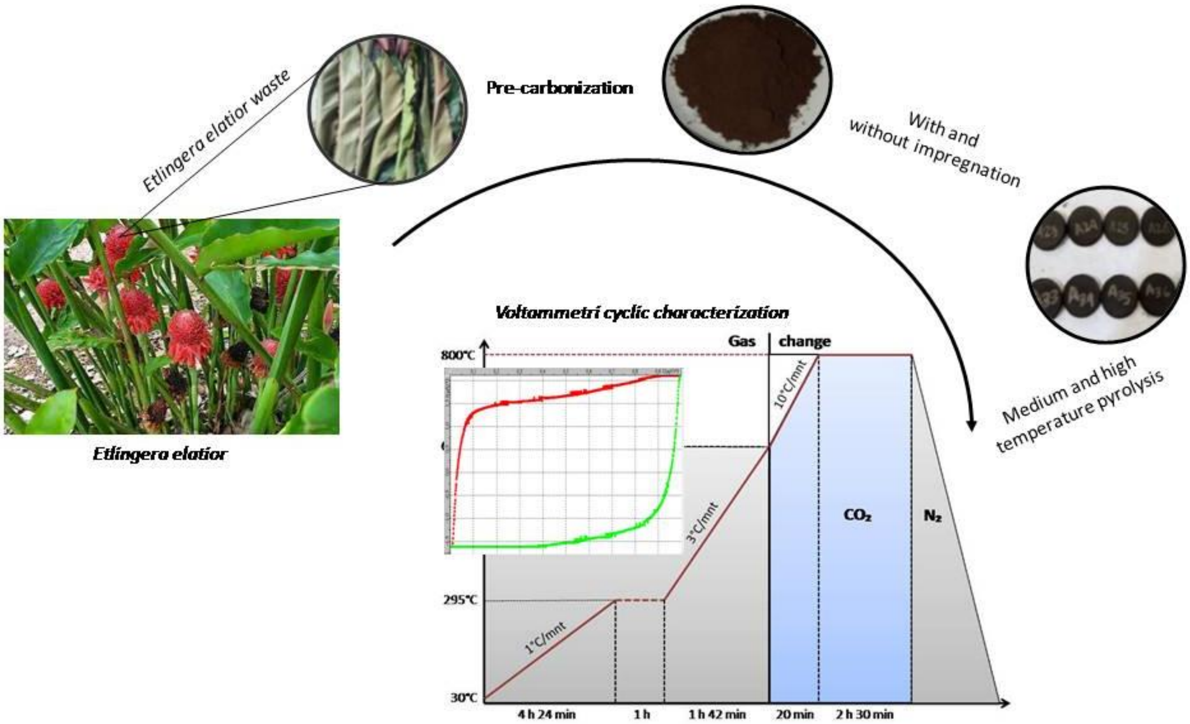

Figure 1. Schematic of preparation of activated carbon monolith derived from *Etlingera elatior*.

The physical properties of the monolith electrodes were characterized such as mass shrinkage, diameter and thickness of carbon pellets before and after pyrolysis, to calculate the percentage of density shrinkage using standard equations. The electrochemical properties were tested by assembling a carbon monolith electrode in the shape of a coin like a sandwich layer. Two symmetrical activated carbon monoliths as material electrodes in 1 M Na_2SO_4 aqueous electrolyte are separated by a duck eggshell membrane which acts as a separator. This test uses the cyclic voltammetry method and galvanostaic charge-discharge (GCD), instrument CV (CV UR Rad-Er 5841) and GCD (CD UR Rad-Er 2018), these instrument were calibrated with VersaStat II Princeton Applied Research, error ± 6.05%. Specific capacitance (Csp, F/g), energy density (E, Wh/kg), and power density (P, W/kg) were evaluated using standard equations [25,26].

3. Result and discussion

The pyrolysis process is a routine step in the formation of carbon electrodes from biomass material by first measuring the mass, diameter, and thickness of the monolith sample before and after the pyrolysis process. Density shrinkage is strongly influenced by chemical and physical activation [27]. The chemical activation process contributes to the formation of the pore structure which is followed by a single step integrated pyrolysis process. Where, the carbonization process in an N_2 gas environment is carried out from a temperature of 30 °C to 600 °C aiming to remove volatile substances such as oxygen, hydrogen and nitrogen [8,28,29]. Followed by physical activation in the CO_2 gas environment

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up to a temperature of 900 °C to produce new pores and the development of pore size which can reduce the density of pellets [30]. The existing micropores are developed to a larger scale (meso and macropores) through a physical activation process [31,32]. The accumulation of this shrinkage was assessed through the density of the samples, as shown in Figure 2.

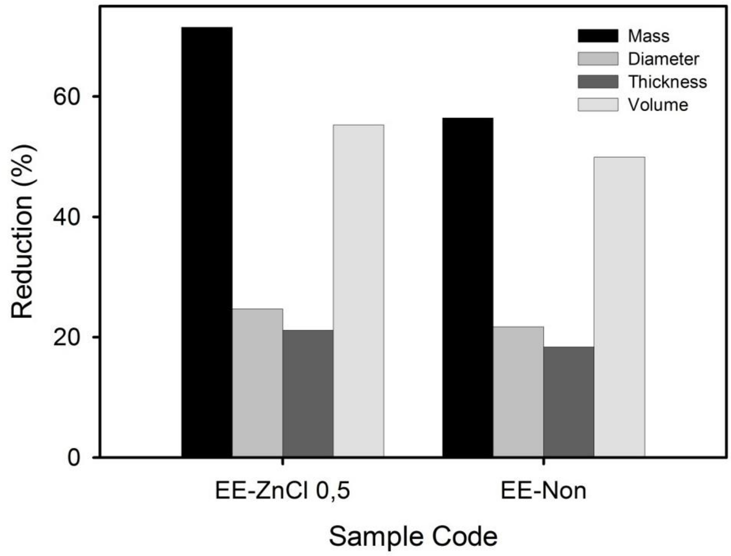

Figure 2. Reduction of mass, diameter, thickness, and volume.

According to Figure 2, the mass, diameter, thickness and volume of the carbon monolith electrodes of the two variations experienced shrinkage. The percentage of mass, diameter, thickness and volume shrinkage of the EE-$ZnCl_2$ 0.5 M sample was 71.49%, 24.68%, 21.15%, and 55.25%, for the EE-Non sample, it was 56%, 43%, 21.7%, 18.35%, and 49.94%. Based on the data on the percentage of shrinkage, chemical activation and physical activation greatly affect the density of the carbon monolith sample [33]. The active oxygen contained in the $ZnCl_2$ activator helps to evaporate volatile elements and impurities in the carbon monolith, thus facilitating the formation of micro pores and meso pores [34,35]. The existing pore structure is developed into larger pores and the formation of new pores through physical activation causes a further decrease in density [36–38].

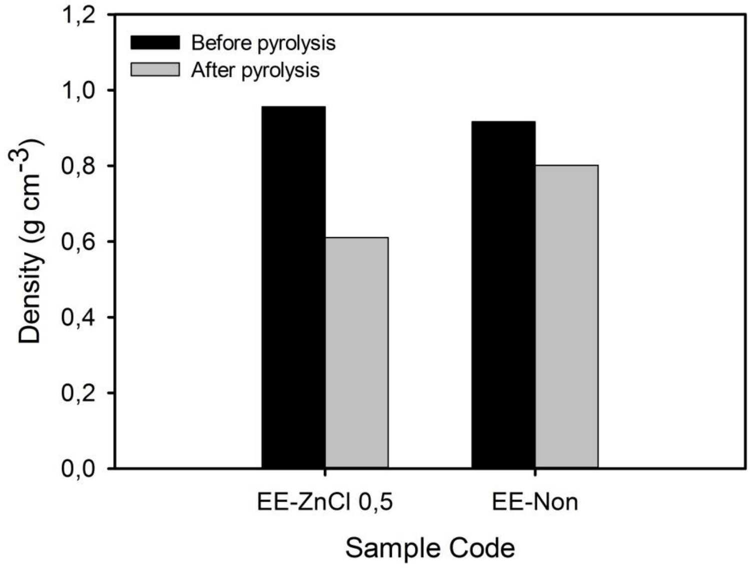

Figure 3. Density of carbon monolith before and after physical activation.

According to Figure 3, it can be seen that the decrease in the density of the carbon electrodes before and after the pyrolysis process for samples with physical chemistry activation and without both decreased respectively. These results are consistent with previous studies using different biomass materials such as durian shell [39] and cassava petiole [40]. The largest density decrease was found in the 0.5 M EE-ZnCl sample, which was 36%, while for the EE-Non sample, it was 13%. From this, it is clear that the role of 0.5 M $ZnCl_2$ activator and physical activation in the formation of the pore structure of the *Etlingera elatior* carbon electrode is clear.

Cyclic voltammetry is the most commonly used technique to determine the electrochemical properties of supercapacitor cell electrodes. The supercapacitor cell was prepared through a two-electrode configuration system consisting of a symmetrical carbon monolith as a conductive material with a 1 M Na_2SO_4 liquid electrolyte as a source of ionic charge. Two carbon electrodes were separated by a separator from the duck eggshell membrane. The CV test was carried out at a voltage range of 0–1 V and a low scan rate of 1 mV/s. The results of the CV test are in the form of a distorted square-shaped graph that comes from the relationship between current and voltage. This shape confirms the general shape of the electrochemical properties of the carbon electrodes derived from the biomass material [19,41].

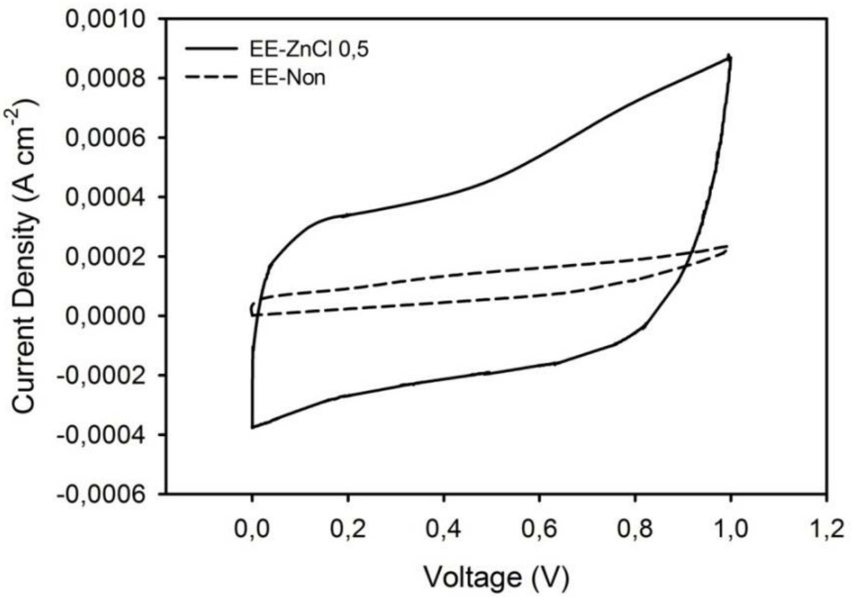

Figure 4. The electrochemical performance of porous carbon in 1 M Na_2SO_4.

Based on the Figure 4, the EE-$ZnCl_2$ 0.5 M sample has a much larger capacitance value than the EE-Non sample. This is due to the effect of adding 0.5 M $ZnCl_2$ activating agent and the physical activation process carried out. Confirmed from the density data discussed above, 0.5 M $ZnCl_2$ activator and physical activation can facilitate the evaporation of impurities other than carbon.

This evaporation creates an empty space in the form of pores, which affects the increase in porosity. The high porosity of the 0.5 M EE-$ZnCl_2$ sample indicates an increasing number of ionic charge contact areas on the electrode surface, resulting in better ion absorption [42,43]. This is evidenced by the specific capacitance value obtained from the EE-$ZnCl_2$ 0.5 M sample of 108 F/g while the chemically unactivated sample can only show a specific capacitance value of 9 F/g. This confirmed that chemical activation with 0.5 M $ZnCl_2$ can increase the specific capacitance of *Etlingera elatior* leaf-based carbon electrodes up to 10-fold from 9 F/g to 108 F/g at a scanning rate of 1 mV/s.

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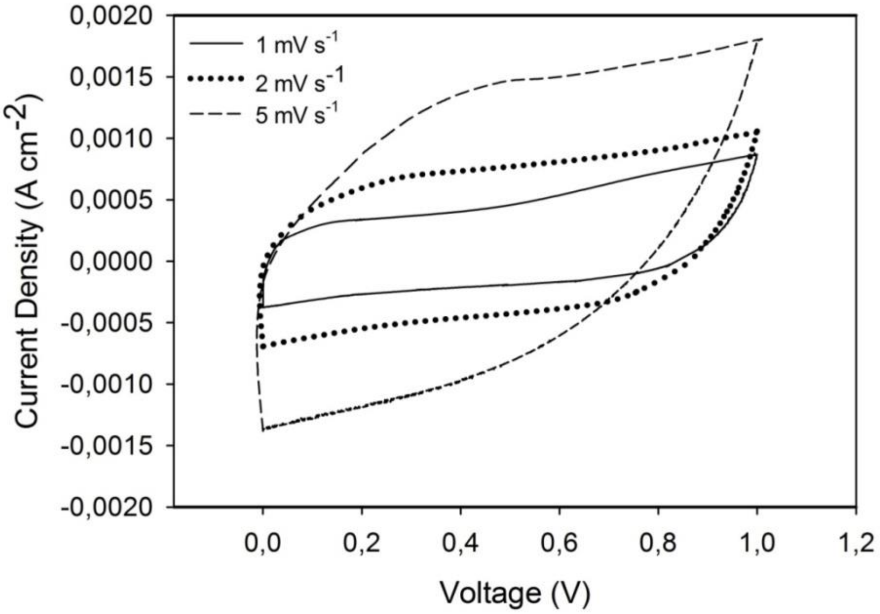

Figure 5. CV curve of EE-$ZnCl_2$ 0.5 M at different scan rates.

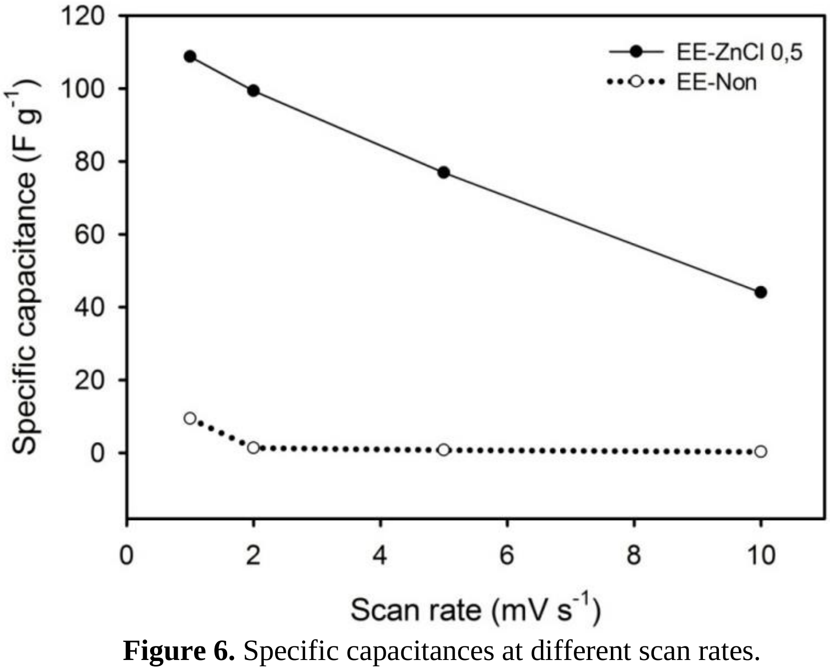

Figure 6. Specific capacitances at different scan rates.

Based on the Figure 5 and 6, the scanning rate greatly affects the specific capacitance value generated. Where the smaller the scanning rate given, the greater the capacitance value. This is because, the time contribution given by the low scan rate takes longer to raise the voltage from 0 V to 1 V. The length of time given can increase the opportunity for the ions from the electrolyte to be completely distributed.

The electrochemical properties of activated carbon electrodes were further characterized using the galvanostatic charge discharge (GCD) method. The GCD graph looks like a distorted equilateral triangle formed from the relationship between voltage and time. This characterization uses a current density of 1 A/g and a scan rate of 2 mV/s.

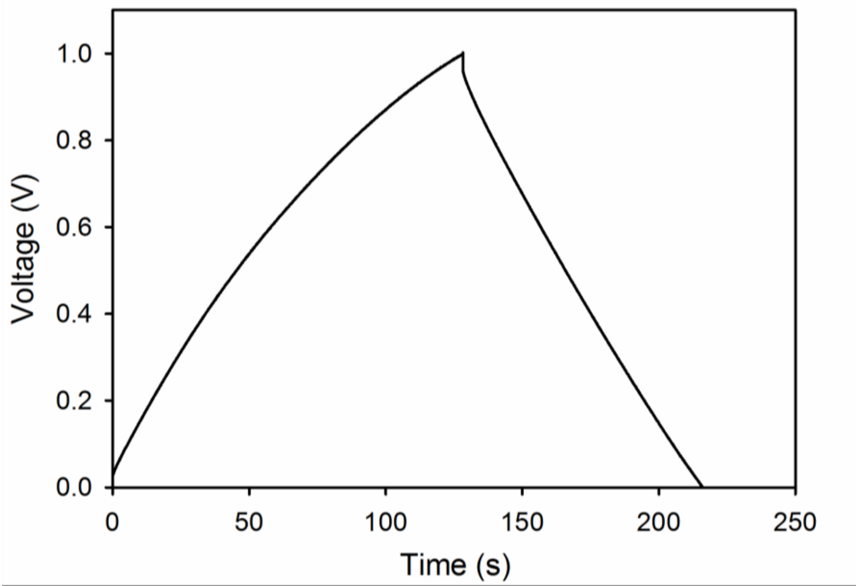

Figure 7. The GCD profile of EE-ZnCl 0.5 M supercapacitor cell.

The Figure 7 shows the GCD curve showing an EDLC type supercapacitor followed by a faradaic reaction due to the influence of the distribution of heteroatom elements. This graph confirms the relatively normal electrochemical properties for the biomass-based carbon electrode base material. The longer charging time allows the electric charge from the decomposed electrolyte to diffuse maximally to fill the pores of the carbon electrode. Optimization of pore filling can increase the specific capacitance value produced, also supported by cyclic voltammetry testing.

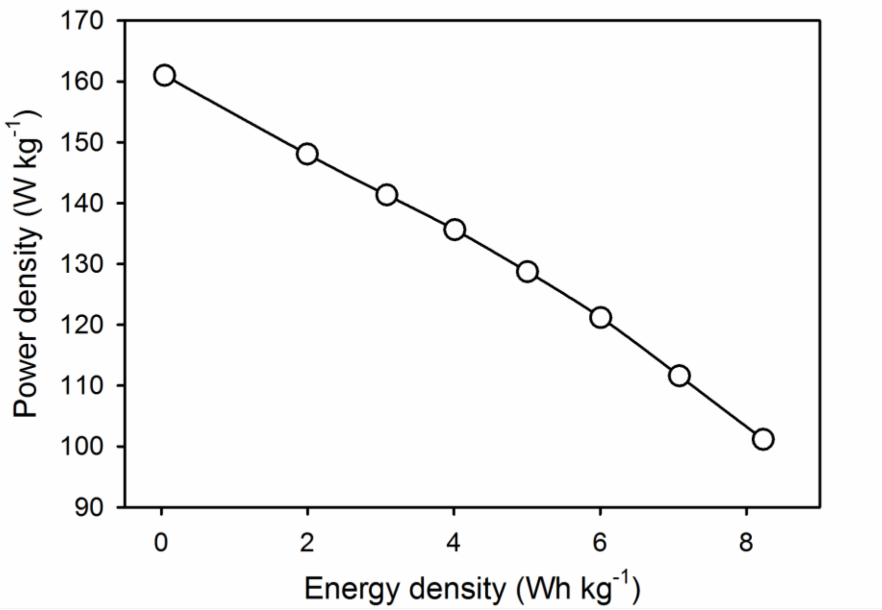

Figure 8. The Ragone of EE-ZnCl 0.5 M samples.

Based on the Figure 8, the GCD test for the EE-ZnCl 0.5 M sample resulted in a specific capacitance of 148 F/g, an energy density of 8.23 Wh/kg, and a power density of 148 Wh^{-1}. The capacitance of *Etlingera elation* leaf-based carbon electrodes without an adhesive material can compete with previous studies of supercapacitors such as tea waste [44], european wood [45], and mangoosten [46,47].

4. Conclusion

Etlingera elatior leaf waste has been successfully prepared and characterized as porous activated carbon for supercapacitor electrodes. Processing that is not complicated and cost-effective because it does not require additional adhesives in its processing can increase the use value of biomass waste. The chemical activation of $ZnCl_2$ in one-stage integrated pyrolysis can increase the electrical charge storage capacity. Electrochemical properties were tested on a symmetrical two-electrode system with Na_2SO_4 electrolyte at a voltage of 0–1 V and a scanning rate of 1 mV/s. Chemical activation with 0.5 M $ZnCl_2$ and physical activation up to 800 °C can increase the electrochemical ability from 9 F/g to 108 F/g. Furthermore, the highest energy density is 8.23 Wh/kg and the power density is 161 W/kg. These results support the high potential in the manufacture of *Etlingera elatior* leaf-based carbon electrodes to improve the electrochemical energy storage performance of supercapacitors.

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