Effect of Carbonization Temperature on the Physical and Electrochemical Properties of Carbon Electrodes from Kepayang Leaves (Pangium Edule Reinw) as Supercapacitor Cells

Pengaruh Temperatur Karbonisasi Terhadap Sifat Fisis dan Elektrokimia Elektroda Karbon dari Daun Kepayang (Pangium Edule Reinw) sebagai Sel Superkapasitor

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ABSTRACT: *The increasing demand for renewable energy resources, supercapacitors are becoming important devices due to their high specific energy and specific power performance. This research focuses on using Kepayang leaves as a basic material for supercapacitor carbon electrodes. This research involves making active carbon from Kepayang leaves through a carbonization process at temperatures of 500, 600, 700, and 800 ℃ for 1 hour with chemical activation using $ZnCl_2$. The characteristics carried out include density analysis, Fourier Transform Infra-Red (FTIR), X-ray diffraction (XRD), and Cyclic Voltammetry (CV). The density analysis results show that with every increase in temperature, the density decreases. Functional groups in the FTIR spectrum show that C bonds are formed, while XRD analysis shows an amorphous structure both before and after pyrolysis. The electrochemical properties of Kepayang leaf carbon show that the diffusion process is getting better as the carbonization temperature is higher. The highest specific capacitance obtained was based on the results of CV 91 F/g at a temperature of 700 ℃.*

Keywords: *Activated carbon; biomass; capacitance; Kepayang leaves; supercapacitor*

ABSTRAK: *Meningkatnya permintaan sumber daya energi terbarukan, superkapasitor menjadi perangkat yang penting karena kinerja energi spesifik dan daya spesifik yang tinggi. Penelitian ini berfokus pada pemanfaatan daun kepayang sebagai bahan dasar elektroda karbon superkapasitor. Penelitian ini melibatkan pembuatan karbon aktif dari daun kepayang melalui proses karbonisasi pada suhu 500, 600, 700, dan 800 ℃ selama 1 jam dengan aktivasi kimia menggunakan $ZnCl_2$. Kerakteristik yang dilakukan meiputi analisis densitas, Fourier Transform Infra-Red (FTIR), X-Ray Diffraction (XRD), dan Cyclic Voltammetry (CV). Hasil analisi densitas menunjukkan bahwa setiap kenaikan temperatur nilai massa jenis mengalami penurunan. gugus fungsi pada spektrum FTIR memperlihatkan adanya ikatan C yang terbentuk, sedangkan analisi XRD menunjukkan struktur amorf baik sebeum maupun sesudah pirolisis. Sifat elektrokimia dari karbon daun kepayang menunjukkan bahwa proses difusi yang berlangsung semakin baik seiring tinggi temperatur karbonisasi. Kapasitansi spesifik tertinggi yang didapatkan berdasarkan hasil dari CV 91 F/g pada suhu 700 ℃.*

Kata kunci: *Karbon aktif; biomassa; kapasitansi; daun Kepayang; superkapasitor.*

1. INTRODUCTION

The production of electric vehicles has increased in the global market due to the growing demand for environmentally friendly and high-energy resources. At the same time, the depletion of renewable energy sources highlights the need for efficient renewable energy storage solutions [1]. Renewable waste such as biomass can be used as a starting material because it reduces raw material costs and reduces the risks of solid waste management. Biomass is an efficient carbon material because this material can be recycled, is environmentally friendly and is widely available [2, 3]. Activated carbon can be made from residual or waste biomass materials, which are alternative raw materials found [4]. Supercapacitors are used as energy storage devices due to their characteristics, namely the specific power value and specific energy value. The specific power value indicates the speed of energy delivery, while the specific energy value reflects how far the supercapacitor can deliver energy on a single charge [5].

Kepayang leaves (*Pangium to success Reinw*) contain flavonoids and saponins, most of which are taken in making activated carbon, namely Kepayang shell contains 40.99% hemicellulose, 70.52% cellulose, 27.88% lignin, and 16.89% pentosan. Kepayang leaves contain 1.38% ash and 5.64% moisture [6].

In previous research, the use of leaves as activated carbon was carried out by Apriwandi, *et al*, (2021) who used biomass from banana leaves with physical activation at temperatures varying from 700-900 °C with the best specific capacitance KOH activation at the highest activation temperature, namely 245 F/g [2]. Erman Taer, *et al* (2022) conducted research using lemongrass leaf biomass as a source with physical activation at a temperature of 800 °C with $Zncl_2$ activation at a concentration of 0.7 M which produces a supercapacitor specific capacitance, namely 256 F/g [7]. Related research was also carried out by Yanna Mao, *et al* (2021) with biomass from cotton leaves using seawater as an electrolyte which showed a very good capacitance of 212 F/g [8]. Similar research has also been conducted by B. Armynah, *et al* (2019) who used bamboo leaves as a material for making activated carbon with physical activation at temperatures of 750, 800, 850, and 900 °C which had the best capacitance at a temperature of 850 °C, which is 60 F/g [9]. Then developed by M. Jayachandran (2021) a supercapacitor made from carbonized bamboo leaves at a high temperature at 500 °C, activated carbon provided the high specific capacitance found, namely 290 F/g [10].

The research carried out above proves that activated carbon biomass made from leaves can be used as a supercapacitor electrode. Therefore, this research focuses on using Kepayang leaf biomass as a basic material for making supercapacitor carbon electrodes. The samples will be analyzed using density analysis, Fourier-transform infrared (FTIR), X-ray diffraction, and Cyclic Voltammetry (CV). It is hoped that this research will provide a solution to the cost problem of making supercapacitors due to the use of expensive materials**.**

2. RESEARCH METHODOLOGY

2.1. Material

Zinc Chloride ($ZnCl_2$), hydrochloric acid (HCl), H_2SO_4 solution, duck eggshells, and distilled water. Dried Kepayang leaves were taken in Soppeng, South Sulawesi.

2.2. Preparation of carbon electrodes

Kepayang leaves were collected, and the leaf veins were separated from the leaf blades and cut into small pieces. Next, the leaves were cleaned with distilled water. The pre-carbonization stage was conducted at a temperature of 225 ℃ for 1 h, followed by ball milling to obtain sample powder measuring 38 µm. The powder obtained was then chemically activated using a 0.3 M $ZnCl_2$ solution with a $ZnCl_2$ to powder ratio of 3:1. Stirring was carried out to obtain homogeneous results using a magnetic stirrer at a temperature of 80 ℃ for 1 hour. The sample was allowed to reach room temperature and neutralized to normal pH, then the sample was dried in an oven at a temperature of 75 ℃ to 100 ℃ for 16 hours. The activated sample was weighed to determine its mass of 0.7 g and molded using a hydraulic press with a pressure of 99.64 kPa or a mass of 10 tons for 5 minutes so that the resulting pellets are dense, strong, and do not break easily. The pellets were then carbonized and physically activated (pyrolysis). The carbonization process uses a furnace at temperatures of 500, 600, 700, and 800 ℃ with N_2 gas for 1 h, while the physical activation was carried out at a temperature of 850 ℃ with CO_2 gas for 1 h.

2.3. Supercapacitor separator preparation

Duck eggshells were cleaned using distilled water, The $CaCO_3$ was dissolved in the egg, and then it was soaked in 1 M HCl to dissolve the $CaCO_3$. As a result, the eggshell released the outer membrane, apart from the eggshell. The membrane was then washed with distilled water so that the pH became neutral, then the membrane was soaked in solution H_2SO_4 1 M for 2 days and the separator was ready to use.

2.4. Supercapacitor cell preparation

Stainless steel which has been shaped according to the diameter of the electrode, cleaned with distilled water, and left to dry until clean. The carbon electrode was immersed in H_2SO_4 1 M for 2 days. A skin membrane was placed between the carbon electrodes as shown in Figure 1.

Figure 1. The arrangement of supercapacitor cell components.

2.5. Characterization

The samples were characterized using density analysis to determine the density of the carbon electrode, XRD to see the crystal structure formed from activated carbon, FTIR-to determine the functional groups formed on the carbon electrode. Physics Cyclic Voltammetry UR Rad-Er 5841 is used to determine the performance resulting from supercapacitor cell electrodes. Capacitance can be calculated with the following Eq. (1) [12].

$$C_{sp} = \frac{Ic - Id}{Sm} \tag{1}$$

Csp is the specific capacitance (F/g), I_c is the charging current (A), I_d is the current during discharge, *S* is the scanning rate (mV/s) and *m* is the mass of the electrode (g).

Figure 2 provides a comprehensive explanation of the entire process of supercapacitor fabrication, from the initial stage to the final stage. This process covered every step, including material preparation, initial processing, and the various technical procedures involved, up to the final assembly of the supercapacitor cell. Each stage was described in detail, illustrating how materials were prepared, and how chemical and physical processes were carried out.

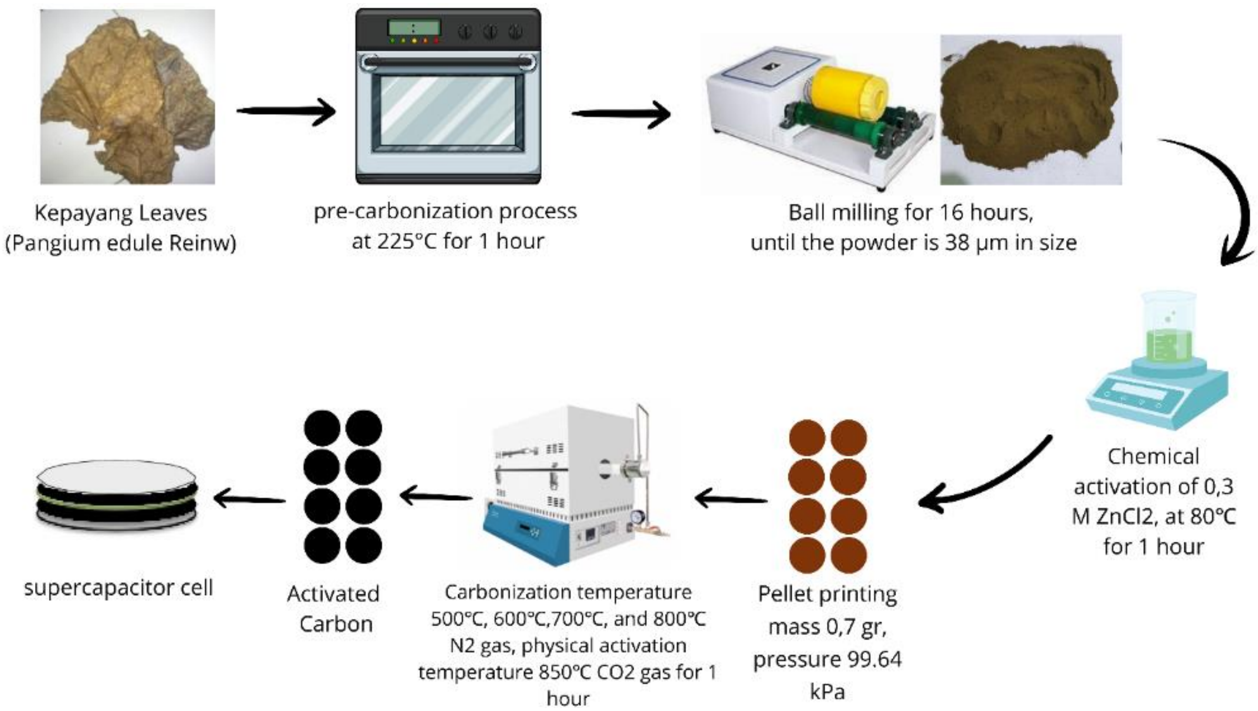

Figure 2. Experimental diagram.

3. RESULTS AND DISCUSSION

3.1. Density analysis

A low-density value will produce a fairly high porosity so that a larger amount of charge is stored. On the other hand, the greater the density, the smaller the number of pores on the electrode so the charge storage will also be smaller [13]. Analysis of the density of carbon electrodes on Kepayang leaves can be seen in Fig. 3.

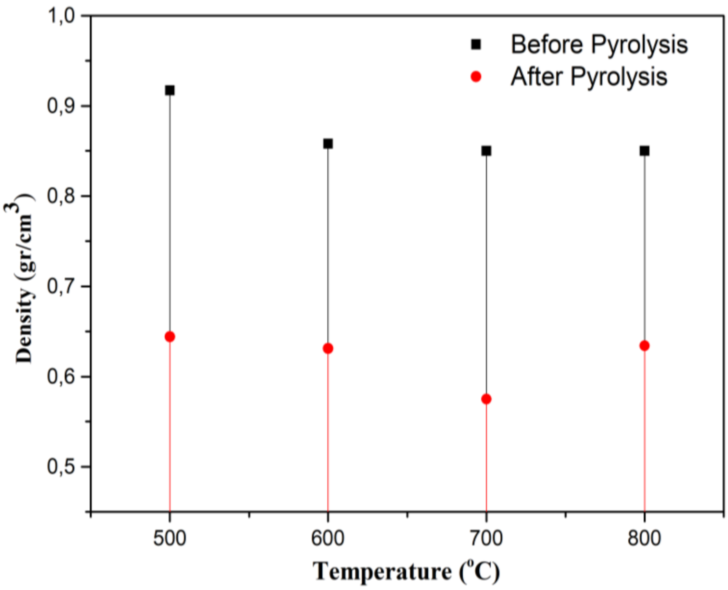

Figure 3. Density before and after pyrolysis.

Density values at each supercapacitor cell electrode before and after pyrolysis with temperature variations of 500, 600, 700, and 800 °C. The density values before pyrolysis are 0.917 g/cm^3, 0.858 g/cm^3, 0.850 g/cm^3, and 0.850 g/cm^3 with a standard deviation of 0.031 g/cm^3. Meanwhile, the density values after pyrolysis are 0.644 g/cm^3, 0.631 g/cm^3, 0.575 g/cm^3, and 0.634 g/cm^3. The highest density loss on the electrode at a temperature of 700 °C is 32.35%, while the lowest density loss is at a temperature of 500 °C, namely 29.74%. The decrease in mass is caused by the evaporation of non-carbon materials and the evaporation of water content from the electrode, which will affect the thickness and diameter values of the pellets. The number of pores formed in the sample affects the density of the carbon electrode of the supercapacitor cell. The pellet-making process also affects the density of the sample due to differences in the uneven surface of the pellets resulting in different thicknesses and diameters even though the mass is the same [14]. The density value after pyrolysis will decrease as the carbonization temperature increases, due to the presence of waste substances that decompose during the pyrolysis process, so the mass and volume of the sample change [15]. Table 1 shows the density values before and after pyrolysis.

Table 1. Comparison of density before and after pyrolysis.

Temperature Variation (°C)	Density (g/cm^3)	
	Before Pyrolysis	After Pyrolysis
500	0.917	0.644
600	0.858	0.631
700	0.850	0.575
800	0.850	0.634

3.2. FTIR analysis

Functional group analysis was carried out using FTIR with a wavenumber of 350-4000 cm^{-1}. The samples analyzed were carbon electrodes before and after pyrolysis, based on the FTIR spectrum of the functional groups formed as in Fig. 4.

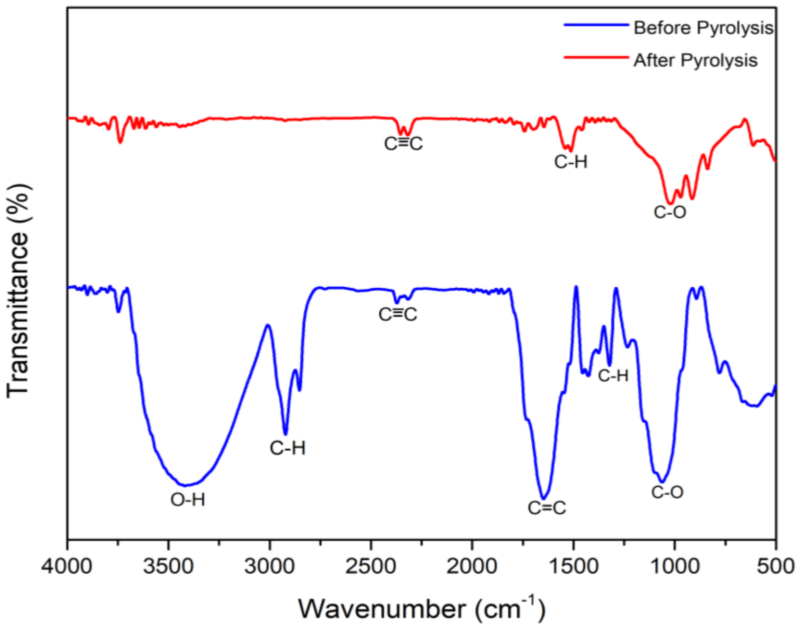

Figure 4. FTIR spectrum of Kepayang leaves before and after pyrolysis.

Based on the FTIR analysis in Figure 4, it produces several functional groups including C–O at wave numbers 1022 cm^{-1} and 1057 cm^{-1}. These functional groups are caused by lignin and appear as a result of an incomplete pyrolysis process. The C–O functional group is a group commonly found in active carbon produced from biomass. The C–H functional group is at a wave number of 1317 cm^{-1}, whereas after pyrolysis it is at a wave number of 1511 cm^{-1}. At a wave number of 1642 cm^{-1}, the C=C functional group is observed, which is a constituent component of an active carbon consisting of layers of graphene. The wavenumber 2310-2366 cm^{-1} shows the presence of the C≡C functional group, while the wave number 2917 cm^{-1} contains the C–H functional group. The main carbon chain forms O–H hydroxyl bonds which are caused by stretching vibrations at wave numbers 3421-3606 cm^{-1}. The abundance of C bonds in functional groups shows a higher carbon content compared to other elements which can be seen in Table 2.

Table 2. Functional groups of carbon leaves of Kepayang.

Functional Groups	Wavenumber (cm^{-1})			Reference
	Absorption Range	Before Pyrolysis	After Pyrolysis	
C–O	1050-1300	1057	1022	[16]
C–H	1340-1570	1317	1511	[17]
C=C	1610-1680	1642	-	[18]
C≡C	2100-2500	2366	2310	[19]
C–H	2850-3000	2917	-	[17]
O–H	3200-3600	3421	-	[20]

3.3. XRD analysis

Analysis XRD aims to determine the structure formed from the supercapacitor cell electrodes. Figure 5 shows the curve of the relationship between x-ray intensity and scattering angle (2θ). Analysis X'pert HighScore Plus was used to determine the carbon phase formed by database matching International Center for Diffraction Data (ICDD).

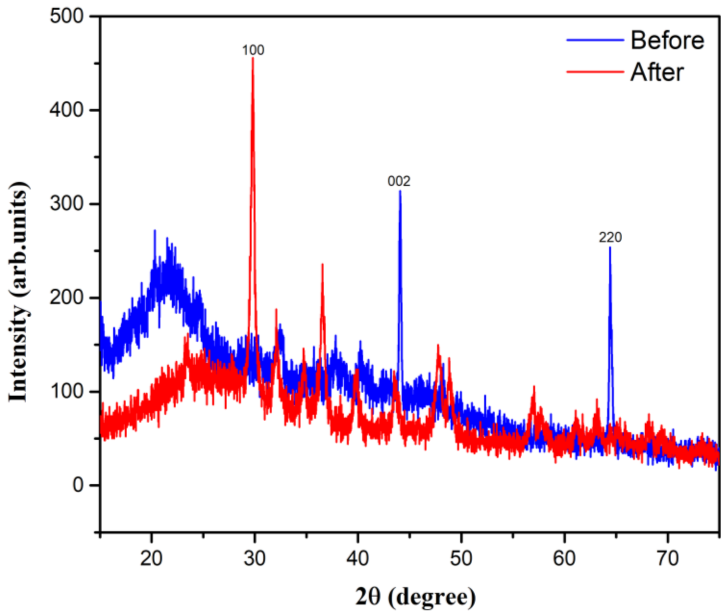

Figure 5. XRD curve of Kepayang leaves before and after pyrolysis.

In Fig. 5, the irregular carbon surface structure causes the formation of pores with varying sizes [21, 22]. Figure 5 shows the XRD light patterns before and after pyrolysis. The structure is an amorphous structure characterized by a wide peak of 16.3º-25.2º and the resulting spectrum appears irregular. The x-ray spectrum before pyrolysis shows that there are two visible diffraction peaks at peak 44.5º in the (002) plane and at peak 64.1º in the (220) plane having ICDD 00-049-1623. Meanwhile, after pyrolysis, several diffraction peaks appear. Notably, there is a peak at 29.5º in the (100) plane and a peak at 42.2º in the (002) plane, as indicated by ICDD 01-072-2091. The peaks were similar in research on agar wood and bamboo leaf carbon, namely at the peak of 34.4º- 67.07º in the (002-220) field with ICDD 01-075-1526 while in the (100) field it was 26.6º- 43.3º ICDD 89-83495 [9, 23]. There are several sharp peaks due to the presence of $CaCO_3$ after pyrolysis and the presence of this compound is extracted from the biomass elements of Kepayang leaves.

3.4. Cyclic voltammetry analysis

Analysis cyclic voltammetry (CV) is used to determine the specific capacitance value on the carbon electrode of the supercapacitor cell and aims to determine the properties of the supercapacitor cell electrode. The scanning rate used to measure electrochemical properties can also affect the size of the resulting *Csp* value. Figure 6 shows the CV curve at temperature variations of 500, 600, 700, and 800 ℃.

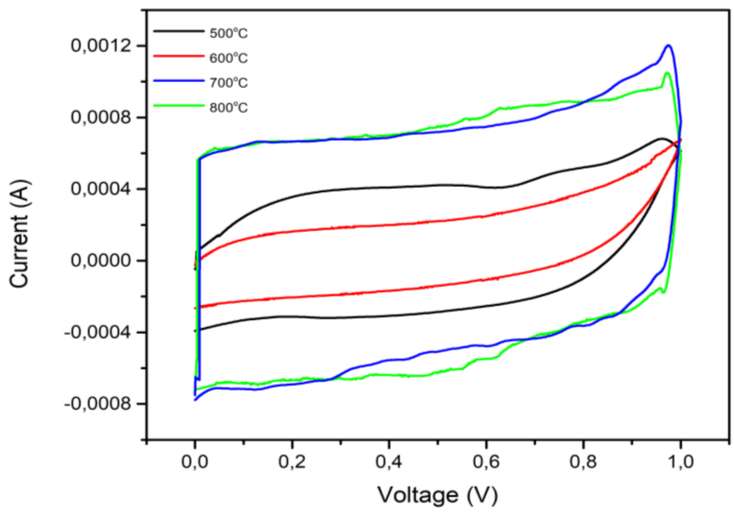

Figure 6. Cyclic Voltammetry curve of supercapacitor cell electrodes.

The voltammogram curve in Fig. 6 shows the relationship between current (A) and voltage (V) in a quasi-rectangular shape with different curve areas. The difference in curve area proves that temperature variations and $ZnCl_2$ activation effect the *Csp* value. Wider curves result in wider capacitance value, and vice versa. This is caused by differences in current during the charge and discharge process which is caused by the charge from the ions [24].

The voltammogram curve identifies the presence of charging current (charge) and emptying (discharge). The current charge and discharge will be larger if it has a large pore size on the electrode. Increased current charge in the positive direction and increased current discharge in the negative direction on the voltammogram curve lead to an enhancement in the capacitive properties of the electrode. This behavior is illustrated in Figure 6, where the improvements in capacitance are clearly demonstrated. The voltage will decrease during the discharge process, allowing ions to escape from the carbon pores of the supercapacitor cell electrode.

As Shown in Table 3, each *Csp* carbonization temperature increases with each increase in temperature [25]. According to Table 3, the temperature of 700 °C has the highest specific capacitance, namely 91 F/g, while the lowest capacitance value at a temperature of 600 °C is 24 F/g. The specific capacitance value obtained shows that ions cannot diffuse perfectly on the surface of the carbon electrode [26]. The specific capacitance value is also influenced by the ion size of an electrolyte [26]. The specific capacitance value of each supercapacitor cell electrode is calculated using equation 1 which is shown in Table 3.

Table 3. Specific capacitance values of supercapacitor cells from Kepayang leaves.

Temperature (°C)	*S* (V/s)	*m* (g)	I_c (A)	I_d (A)	C_{sp} (f/g)
500	0.002	0.0100	0.000413	-0.00029	35
600	0.002	0.0075	0.000221	-0.00014	24
700	0,002	0.0075	0.000740	-0.00063	91
800	0.002	0.0075	0.000716	-0.00031	69

4. CONCLUSION

The physical properties of the Kepayang leaf carbon electrode show that with each increase in temperature the density value decreases and the functional groups formed show the presence of C bonds and produce an amorphous structure both before and after pyrolysis. Meanwhile, the electrochemical properties of Kepayang leaf carbon show that the diffusion process is getting better as the carbonization temperature is higher. The maximum specific capacitance value of the supercapacitor cell electrode from Kepayang leaves is 91 F/g at a temperature of 700°C. Based on these results, carbon electrodes from Kepayang leaves have good performance for use as energy storage devices and are one of the materials with the most potential for use as supercapacitor cells.

Acknowledgment

Thank you to the supervisors who have assisted in this research, thank you to the Head of the Materials and Energy Laboratory, Faculty of Mathematics and Natural Sciences, Hasanuddin University, the Head of the Materials and Energy Laboratory, Faculty of Mathematics and Natural Sciences, Riau University for allowing using Lab in sample making.

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