Effect of physical activation time on the preparation of carbon electrodes from pineapple crown waste for supercapacitor application

by Rika Taslim

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Abstract.The effect of physical activation time in production of activated carbon electrodes from pineapple crown waste on the physical and electrochemical properties of supercapacitor cells has been demonstrated in this study. The samples were activated in the CO_2 gas environment at a temperature of 900 ˚C with the activation time varied to 1.5; 2; 2.5 and 3 hours. Physical properties testing showed the activation time of 2.5 hours is the optimum activation time in the producing of carbon electrodes from pineapple crown waste. The optimum conditions are indicated by the minimum condition on the density and microcristalline height, and the maximum condition on specific surface area and carbon content, so it produce higest specific capacitance for supercapacitor cells. The optimum specific capacitance was found as high as 134 Fg^{-1}. As a complement also has been analyzed the appearance of the surface morphology of the sample where the electrode is composed of carbon nanofiber with an average diameter of 90-164 nm.

1. Introduction

Electrochemical double layer capacitors (EDLC) are energy storage devices consisting of porous carbon electrodes, electrolyte, separator and current collectors [1]. Energy storage occurs because of the presence of ion and electron layers formed in the micro pore of the carbon electrode. The more ion pairs and electrons that form the greater the energy that can be stored. The formation of ion and electron pairs is affected by the number of micro pores available in an electrode. Micro pores electrodes are relate to the materials and activation process. Power is the main factor in the performance of EDLC devices other than energy. Power is related to the speed of ion diffusion process into the pores of the electrode to form ion-electron pairs. The pace of ion diffusion is clearly related to the shape of the electrode constituent material and pore size. In the last decade, the shape of electrode constituent materials becomes a fairly extensive study [2]. Various shape of electrode constituent materials have been reported such as cubic [3,4,5], spherical, flat, fiber [6,7] provided in nano or micro sizes. The shape of fiber is one of the most developed forms due to several advantages such as hollow pores and good electrical conductivity [7]. The selection of the original material in the prepare of carbon fiber is one of the interesting research focuses. Production costs are a major consideration in the selection of original material. Biomass promises low production costs, abundant availability and

also being a by-product in the agricultural industry [8]. Some biomass materials as originating materials for the prepared of carbon fiber electrodes that have been reported, such as banana stems [9], coconut husk [7], oil palm empty fruit bunches [10], etc. Different biomass materials provide different sizes of fiber diameters. Pineapple crowns are one of the biomass that contains fiber. The fiber sizes vary in nanoto micro meters.Indonesia is one of the pineapple producing countries with production reaching 1.73 ton in the year 2015 [11]. In this research, a simple method is used to produce carbon fiber electrodes from pineapple crown waste as supercapacitor electrodes. Carbon electrodes are provided without additional adhesives and are activated conventionally using KOH and CO_2 gas. The results showed that the carbon electrode from pineapple crwon produced supercapacitor cells as high as 134 F g^{-1} and found to contain dominant carbon fiber with a diameter size an average of 90-164 nm.

2. Experimental Method

2.1. Preparation of carbon electrode

The biomass used as raw material in this study is pineapple crown. The production of activated carbon from the pineapple crown uses the method previously reported [12]. Pre-carbonization was carried out by heating at a temperature of 250℃ for 2.5 hours. The pre-carbonization process will cause the sample color to be dark-brown and self-adhesive in the sample will appear. The sample is smoothed with a ball milling instrument. The ball milling process is carried out with the aim to produce finer the dark-brown pineapple crown powder. The dark-brown pineapple crown powder was activated using KOH activator with a ratio of 1:1. The sample powder is converted into a monolithic or pellet form by using a Hydraulic Press system [13]. Pyrolysis processes such as carbonization and physical activation are carried out using a one-step integrated method [14]. Carbonization begins with the flow of nitrogen gas (N_2) with a gas flow rate of 1.5 L min^{-1} at a temperature of 600℃ and followed by physical activation by flowing CO_2 gas at a temperature of 900℃. Physical activation time were varied at 1.5, 2, 2.5 and 3 hours. Based on this time variation, each sample is labeled AC-1.5, AC-2.0, AC-2.5 and AC-3.0. Then carbon pellets were wash until the washing water becomes neutral [15]. The preparation of supercapacitor cells in two-electrode system consisting of carbon electrodes, separator, electrolytes and current colectors [1,16]. Separator used is the duck eggshell membrane [17] and H_2SO_4 solution as electrolyte [18].

2.2. Characterization of physical and electrochemical properties.

Measurement of physical properties of pineapple crowns carbon electrodes included of density, crystalline structure, surface morphology and component elements of carbon electrodes while the characterization of electrochemical properties included of capacitive properties of supercapacitor cells. Density is calculated by measuring the mass and volume of the electrode. Analysis of surface morphology and element content of carbon electrodes using Scanning Electron Microscophy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) methods with Jeol JSM 6510 LA instrument. The crystallinity is caraterized by X-ray Diffraction method with X-Pert Powder panalyticsl instrument. Capacitive properties testing using the Cyclic Voltammetry method with the Physics CV UR Rad-Er 5841 which is controlled by cyclic voltammetry CV v6 software.

3. Result and Discussion

3.1. Density Analysis

Comparison of density before and after pyrolysis for carbon electrode samples can be seen in Figure 1. Figure 1 is shown pre-carbonized sample before the pyrolysis process have an average density of 0.932 gcm^{-3}. After pyrolysis process shows the effect of activation time on the density of carbon electrodes, where the density decreases with the addition of activation time (from 0.924 gcm^{-3} at a AC-1.5 sample to 0.625 gcm^{-3} at a AC-2.5 sample). This density reduction is affected by the release of pollutant components such as volatiles which cause pore formation. An increasing in activation time causes the more effective reaction between carbon and CO_2 so that the density decreases. After the addition of activation time of 2.5 hours the sample had an increase in density of 15.2%. This is indicated by at 3

hours, the sample has suffered decay to the particles and the pores are not formed in optimum condition. All the density data, the lowest density occurs at 2.5 hours activation time, this 2.5 hours activation time is chosen to be the best activation time to produce activated carbon from the pineapple crown waste.

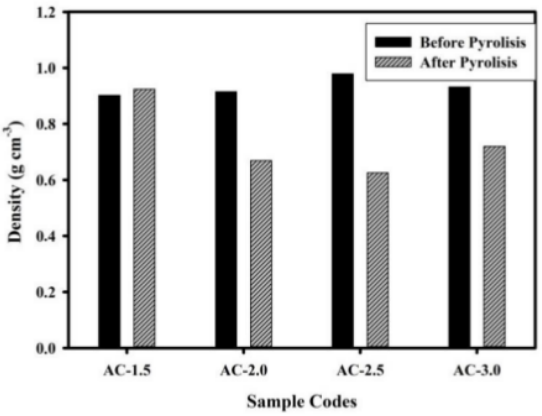

Figure 1. Density of carbon electrode before and after pyrolisis process

3.2. Scanning Electron Microscopy Analysis

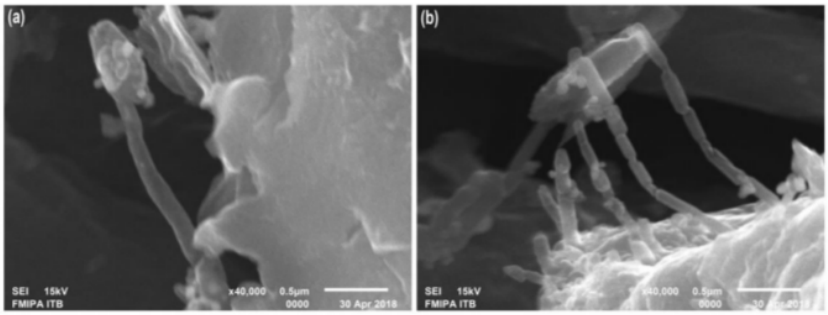

Figure 2.SEM micrographswith a magnification of 40000 times for a) AC-2.0; b) AC-2.5

Characterization of Scanning Electron Microscopy is used to determine the morphological surface structure of activated carbon electrode. Morphological surface structure of pineapple crown activated carbon for AC-2.0 and AC-2.5 samples with a magnification of 40,000 times are shown in Figure 2. Figure 2 (a) shows the results of SEM AC-2.0 carbon electrodes have pores between particles that look elongated and irregular. In addition, there is also a presence of nanofiber structures, where the average fiber diameter range of 164 nm. Figure 2 (b) presents the SEM results for AC-2.5 with larger pores between particles. The resulting nanofiber structure is more clearly visible with a relatively smaller size with a sample diameter in range of 83 to 90 nm. The size diameter in this study are same carbon fiber electrode with the diffrent biomass such as banan stems in range 42-131 nm [9]. The particles formed are affected by the activation time, increasing activation time results in a finer fiber size. This is indicated because the addition of activation time provides a longer chance for the process of carbon and CO_2 reactions so that the carbon chain termination will increase. Termination of the

carbon chain results in cracks and faults in larger particles, increasing activation time also causes faster particle movements resulting in larger collisions between particles. Collisions between particles will cause carbon particles to split into smaller parts.

3.3. Energy Dispersive X-ray (EDX) Analysis

The EDX Characterization was carried out to prove the presence of carbon on the electrode sample made from pineapple crown waste. EDX analysis strengthens the data that has been obtained from the SEM and XRD characterization that will be analized in the next subsection. EDX analysis produces data in a percentage of the quantity of elements of activated carbon sample. Based on the data in Table 1, the element quantities of the carbon electrode consist of carbon (C), oxygen (O), magnesium (Mg), silicon (Si), phosphorus (P), Potassium (K) and calcium (Ca). The element quantities are dominated by carbon with a range of 92% to 94%. These results prove that the samples dominated by carbon as desired. The carbon content obtained in this study is also similar to the previously reported carbon content with different carbon materials, such as from durian [19] and palm date [20].

Table 1. The chemical content for AC-2.0 and AC-2.5 samples

Contents	AC-2.0		AC-2.5	
	Mass %	Atom %	Mass %	Atom %
C	87.99	92.01	91.7	94.62
O	8.24	6.47	5.5	4.26
Mg	1.19	0.61	0.98	0.5
Si	0.53	0.24	0.24	0.1
P	0.23	0.09	0.19	0.08
K	0.91	0.29	0.65	0.21
Ca	0.92	0.29	0.73	0.23
Totals	100%	100%	100%	100%

In addition to carbon, the oxygen element also dominates the sample. The presence of oxygen elements is obtained from the CO_2 gas at activation process. The oxygen element has the second highest percentage after Carbon element, the cause is indicated by the carbonization of oxygen in the carbon samples which do not decompose completely or the bond occurs in the activation process. Carbon element quantity in AC-2.5 sample is greater than AC-2.0 sample. Increasing activation time will cause the quantity of content other than carbon to decompose more, so that the carbon element produced be higher.

3.4. X-ray diffraction (XRD) Analysis

X-ray diffraction is a method to determine whether a material is amorphous or crystalline. The XRD pattern of carbon samples derived from pineapple crown biomass with a activation time variation of 2 hours and 2.5 hours is shown in Figure 3. Characterization show that there are two broadening peaks with a diffraction angle (2θ) in range of 10˚to 100˚. Determination of diffraction angle for each peak was done by using Microcal Origin software. Data is fitted by the lorentzian distribution function. From the fitting data obtained the diffraction angle, peak height and peak width. The AC-2.0 sample has two broadening peaks at a 2θ angle of 25.108˚and 44.214˚ and it are correspond to the scattering plane (002) and (100). While the AC-2.5 sample has a 2θ angle of 24,749˚and 43,768˚.

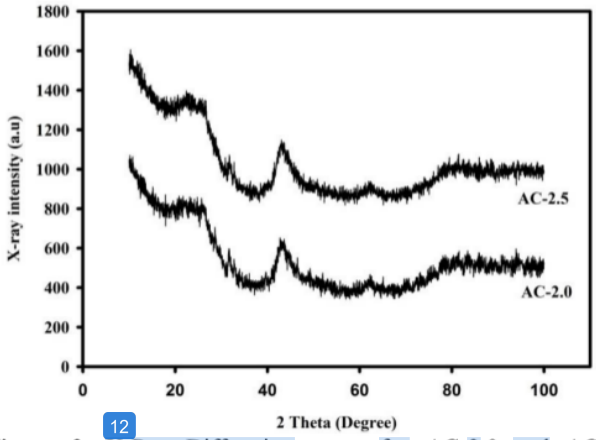

Figure 3. X-Ray Diffraction curve for AC-2.0 and AC-2.5 samples

Table 2 shows that the 2θ angle undergo a shift for samples that were activated at 2 and 2.5 hours. The 2.5 hour activation time has a 2θ angle (002) and (100) close to a universal peak which is 24˚ and 44˚ for carbon peak compared to a 2 hour activation time. This analysis is indicated because a 2.5 hour sample has a higher carbon purity compared to a 2 hour activation time such as EDX data which it discussed earlier. The microcrystalline height data (L_c) shows the existence reguler value based on the activation time variation. The L_c on the AC-2.0 sample is greater than the L_c for the AC-2.5 sample. The relationship of the microcrystalline height (L_c) of graphite in the carbon electrode to the specific surface area is given by the empirical formula $SSA_{xrd} = 2/(\rho_{xrd} L_c)$, where ρ_{xrd} = X-ray density given with the formula $\rho_{xrd} = \{d_{002\ (graphite)} / d_{002}\}\rho_{(graphite)}$[21, 22]. Based on the empirical formula, to produce a higher surface area of carbon electrodes, a smaller interlayer spacing (d_{002}) and microcrystalline height (L_c) are needed. Based on the equation previously, specific surface area obtained for 2 hours sample was 1484.67 m^2g^{-1} and specific surface area for AC-2.5 hours was 1856.54 m^2g^{-1}. This specific surface area aresimilar as the electrode surface area of other materials, such as cococnut husk and cassava peel waste, which are 28982 m^2 g^{-1} [7] and 1352 m^2 g^{-1} [23], respectively. The microcrystalline width (L_a) data shows a significant difference in the variation of activation time in each sample. The AC-2.0 activation time shows a L_a of 25.5839 Å while the AC-2.5 sample has a L_a of 39.8201 Å, where the longer the activation time is the greater L_a. Based on this data it can be concluded that the La is influenced by the length of time of sample activation.

Table 2. Diffraction angle (2θ), interlayer spacing (d), microcrystallinity height (L_c) and microcrystallinity width (L_a) for AC-2.0 and AC-2.5 samples

Sample codes	$2\theta_{(002)}$ (˚)	$2\theta_{(100)}$ (˚)	$d_{(002)}$ (Å)	$d_{(100)}$ (Å)	L_c (Å)	L_a (Å)
AC-2.0	25.108	44.214	3.544	2.047	14.195	25.584
AC-2.5	24.749	43.768	3.594	2.067	13.689	39.820

3.5. Electrochemical PropertiesAnalysis

The electrochemical Measurement of supercapacitor cells was carried out using the Cyclic Voltammetry (CV) method based on two electrode systems in 1M H_2SO_4 electrolyte. Figure 4 is a voltammogram data that shows the relationship between C_{sp} (Fg^{-1}) to voltage (V). The scan rate given is 1 mVs^{-1} at a voltage of 0 to 0.5 V [24]. The selection of the scanning rate of 1 mV s^{-1} is based on the assumption that ion diffusion is more evenly distributed throughout the surface and pores of carbon

electrodes so that the ion pair formation be more perfect, which eventually results in greater specific capacitance [25].

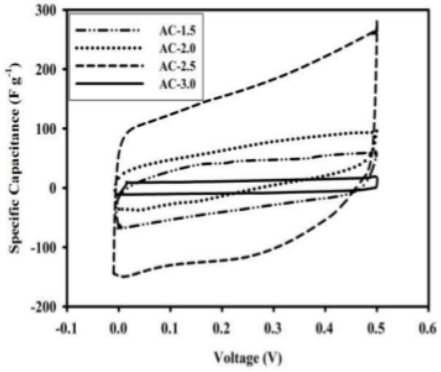

Figure 4. CV curve for all samples

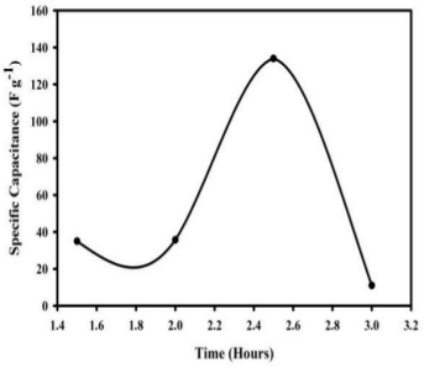

Figure 5. Correlation Specific capacitance with activation time

All samples have specific capacities of 35 Fg^{-1}, 36 Fg^{-1}, 134 $Fg^{-1}$and 10 $Fg^{-1}$for samples of AC-1.5, AC-2.0, AC-2.5 and AC-3.0, respectively. The AC-2.5 sample is the sample with the highest specific capacitance, while the AC-3.0 sample is the sample that has the smallest specific capacitance. The maximum specific capacities produced in this study are almost the same as the specific capacitance with different biomass materials that have been studied, such as rubber wood sawdust with specific capacitance of 150 Fg^{-1}[26] and cow dung with specific capacitance of 124Fg^{-1}[27]. The difference in the specific capacitance is strongly influenced by the density of the activated carbon electrode. Density is inversely proportional to porosity, it mean that the greater the density of the sample, the porosity will be smaller. Porosity is directly proportional to the surface area, the greater the porosity of the sample, the greater the surface area. High surface area gives the opportunity for more ions to experience diffusion resulting in maximum capacitive properties. The results obtained of the AC-2.5 sample has the smallest density and the largest specific capacitance value, while the AC-3.0 sample has the largest density and the smallest specific capacitance. Specific capacitance is also influenced by the addition activation time which be seen in Figure 5. In the figure 5, it can be seen that the longer the activation time will increase the specific capacitance, but when the 3 hour activation time will decrease in the specific capacitance. The 2.5 hour is the activation time with the highest specific capacitance, this is because at that time the meso and micro pores formed are very good. The combination of micro pore and meso pore which more than 2.5 hours develops into larger pores and became macro pores. The decreasing of specific capacitance for AC-3.0 is indicated the 3 hour activation time caused enlargement to micro and mesopore into macro pores.

Conclusion

The production of activated carbon electrodes from pineapple crown waste based on variation of activation time for supercapacitor device has been succesfully done. The optimum activation time was found at 2.5 hours which shown by the good physical and electrochemical properties. The good physical properties inclued of lowest density, fiber structure of surface morfology, highest carbon content and larger specific surface area. This good physical properties supports to found an excelent electrochemical properties with the highest spesific capacitance as high as 134 $F\ g^{-1}$.

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