The Effect of Particle Size on the Performance of Electrode Supercapacitor based on Pepper (Pipper Nigrum) Shell Activated Carbon

W B Kurniawan, A Indriawati and D Marina
Department of Physics, University of Bangka Belitung

Corresponding author’s: widodokurniawan1@gmail.com

Abstract. The synthesis of pepper shell activated carbon-based supercapacitor electrodes to determine the effect of particle size on electrode performance has been done. The pepper shell activated carbon was synthesized using HCl activator with a ratio of 1: 4 (W/V) and then activated physically in an inert condition at 600°C. The characteristics of activated carbon samples were carried out using SEM and BET. SEM results show that carbon has a porous morphology with radius size of absorption pores of 130.0 nm and 121.9 nm respectively for particle size samples of 74 microns and 149 microns. The results of cyclic voltammetry (CV) to find out the value of specific capacitance at a scan rate of 50 mV/s showed a amount of 0.037 F/g and 0.0075 F/g for samples 74 and 149 microns. The results showed the influence of particle size on the performance of supercapacitor electrodes.

1. Introduction
An innovation in the field of energy storage media which the last few decades to interest develop is the supercapacitor. Supercapacitors have several advantages compared to batteries such as high power density and long life cycles [1], also high capacitance values (in range farad) [2]. The supercapacitor consists of electrodes, electrolytes, separators, and current collectors. Thus, the energy storage capability of a supercapacitor electrode is strongly influenced by pore size, surface area and the shape of the electrode particles [3].

One of the electrode materials that is currently the main attraction for researchers is the activated carbon from biomass waste because it's relatively inexpensive and easy to obtain. Biomass waste research has been carried out, for example, from banana peels [3], candlenut shell [4], kluwak shell (Pangium Edule) [5], water chestnut [6], wool felt [7], peanut shell [8]. This research focuses on the effect of particle size of activated carbon from pepper shell waste on the electrode ability of supercapacitors besides that this research is a continuation of previous study [9].

2. Experimental
The synthesis of pepper shell activated carbon as an electrode of supercapacitors was carried out using the method previously reported [9]. The first stage is that pepper shell waste is dried in the sun to dry. After that, roasting will become carbon (charcoal). Then milling using agate mortar until smooth and continued with sieving using sizes 100 (149 microns) and 200 mesh (74 microns). The next stage is chemical activation using HCl activator with a volume ratio of HCl to carbon mass of 1: 4 and stirring using magnetic stirrer until the homogeneous mixture is allowed to remain for 48 hours. Then proceed with washing using distilled water to neutral pH.
The last stage of the synthesis of pepper shell waste activated carbon is physical activation in an inert condition which is fed by N2 gas with a temperature increase of 200°C/hour to reach 600°C and holding time for 3 hours.

The morphology and composition of pepper shell activated carbon was determined using a Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX) using a HITACHI SU-3500 brand. While the sample pore size was carried out using BET the Quantachrome Nova 4200e brand. The electrochemical properties of supercapacitor electrodes from pepper shell activated carbon were carried out using the cyclic voltammetry (CV) method to determine the specific capacitance values based on the resulting voltammogram data and standard equations as previously reported [6].

3. Result and Discuss

3.1. Characteristic of physical property
The morphology of samples of pepper shell activated carbon for 100 mesh and 200 mesh is shown in Figure 1, which shows that pepper shell activated carbon samples have porous morphology. However, for the 200 mesh sample (Figure 1(c) and (d)) the pore wall will become increasingly invisible due to breaking into smaller particles.

![SEM result data](image)

Figure 1. SEM result data; (a) and (b) samples of 100 mesh active carbon shell; (c) and (d) 200 mesh pepper activated carbon samples

The composition contained in samples of pepper shell activated carbon was analysed based on EDX data, as shown in Table 1. The percentage of carbon dominates in samples both 1 and 2. However, the percentage of carbon in sample 2 tends to be less than sample 1. It is possible for the presence of silica elements in sample 2 which tends to become ash so that it can cause reduced carbon content.
Table 1. EDX analysis results for sample 1 and sample 2

| Element | Sample 1 (100 mesh) | Sample 2 (200 mesh) |
|---------|---------------------|---------------------|
|         | % Weight | % Atom | % Weight | % Atom |
| C K     | 71.57    | 81.11  | 70.76    | 79.13  |
| O K     | 19.65    | 16.72  | 22.53    | 18.91  |
| Si K    | -        | -      | 1.12     | 0.54   |
| Cl K    | 0.89     | 0.34   | 0.98     | 0.37   |
| Ni K    | 7.88     | 1.83   | 4.61     | 1.05   |
| Total   | 100.00   | 100.00 | 100.00   |        |

The surface area of adsorption, pore-volume and pore radius based on BET data are shown in Table 2. Based on the data in Table 2, it can be seen that the two samples have pores belonging to the mesopore sizes [10]. Besides that, for sample 2, it tends to have a much larger surface area and also the pore size is more larger than sample 1.

Table 2. BET results of pepper shell activated carbon samples

| Types of Sample | Surface Area (m²/g) | Pore Volume (cc/g) | Pore Radius (nm) |
|-----------------|---------------------|-------------------|------------------|
| Sample 1 (100 mesh) | 0.395   | 0.014  | 121.8 |
| Sample 2 (200 mesh) | 0.880   | 0.028  | 130.0 |

3.2. Electrochemical properties of samples

The electrochemical properties of pepper carbon-based supercapacitor electrodes were analyzed by cyclic voltammetry in the voltage range 0.0 - 0.5 volts. Based on the voltammogram data, as shown in Figure (2) shows that all samples form a rectangle. Similar CV results usually represent the capacitive nature of a carbon-based electrode [11]. The higher the difference between the charge (Ic) and discharge (Id) currents generated from the voltammogram graph shows, the greater the value of the capacitance produced [12].

![Cyclic Voltamogram](image)

**Figure 2. Cyclic voltametry of sample**
The magnitude of the specific capacitance value of pepper shell activated carbon-based electrodes can be determined based on CV results data using the equation at reference [6]. The amount of specific capacitance for each sample is shown in Table 3. The value of specific capacitance in sample 2 has a much greater value than in sample 1. This is because sample 2 has a much larger adsorption surface area and a much larger pore size small so that the pore size affects the specific capacitance value of the supercapacitor electrode which also represents the ability of the supercapacitor electrode synthesized from pepper shell waste.

| Types Sampel | Massa Electodes (g) | Ic (A) | Id (A) | Cₛₚ (F/g) |
|--------------|---------------------|--------|--------|-----------|
| Sample 1 (100 mesh) | 0,12 | 0,000058 | 0,000013 | 0,0075 |
| Sample 2 (200 mesh) | 0,12 | 0,000986 | 0,000763 | 0,037167 |

4. Conclusion
Based on the results of the study showed that the synthesis of electrode capacitors based on pepper shell has a porous structure. Specific capacitance values that represent the ability of the supercapacitor electrode to store the charge are produced at 0.037 F/g for samples with 200 mesh particle size and 0.0075 F/g for 100 mesh sized particles. So that particle size greatly affects the ability of the supercapacitor electrode to store charge.

Acknowledgement
This research partially funded by Ministry of Research, technology and higher education, University of Bangka Belitung, and USAID through the SHERA program - Centre for Development of Sustainable Region (CDSR). In the year 2017-2021 CDSR is led by Center for Energy Studies - UGM.

References
[1] Fang L I, Junjun S H I and Xue Q I N 2010 Synthesis and supercapacitor characteristics of PANI /CNTs composites 55 1100–6
[2] Widiatmoko P, Devianto H, Nurdin I and Yandra R E 2016 The Effect of Carbon Nanotube Composite Addition on Biomass-Based Supercapacitor J. Eng. Technol.Sci 48 597–613.
[3] Taer E, Taslim R, Aini Z, Hartati S D and Mustika W S 2017 Activated Carbon Electrode from Banana-Peel Waste for Supercapacitor Applications AIP Conference Proceedings vol 040004.
[4] Nurdiati D A 2015 Sintesis Komposit PANI/Karbon dari Tempurung Kemiri (Aleurites moluccana) sebagai Elektroda Kapasitor J. Fis. Unand 4 51–7.
[5] Habibah M D, Rohmawati L and Setyarsih W 2016 Variasi Holding Time Suhu Aktivasi Karbon Aktif dari Tempurung Kluwak (Pangium Edule) sebagai Elektroda pada Superkapasitor 05 19–22.
[6] Zulkifli, Awitdrus and Taer E 2018 The Preliminary Study of Utilization of Water Chestnut as Supercapacitor Electrode Using Steam Activation J. Aceh Phys.Soc 7 30–4.
[7] Pina A, Amaya A, Marcuzzo J, Rodrigues A, Baldan M, Tancredi N and Cuña A 2018 Supercapacitor Electrode based on Activated Carbon Wool Felt C 4 24.
[8] Xiao Z, Chen W, Liu K, Cui P and Zhan D 2018 Porous Biomass Carbon Derived from Peanut Shells as Electrode Materials with Enhanced Electrochemical Performance for Supercapacitors 13 5370–81.
[9] Kurniawan W B, Indriaawati A, Marina D and Taer E 2019 The Potential of Pepper Shell (Piper Nigrum) for Supercapacitor Electrodes J. Pendidik. Fis. Indones. Al-Biruni 08 109–16.
[10] Sing K S, Haul R A, Pierotti R . and Siemieniewska T 1985 International Union of Pure Commission on Colloid and Surface Chemistry Including Catalysis * Reporting Physisorption Data for Gas / Solid Systems with Special Reference to the Determination of Surface Area and
Porosity *Pure Appl. Chem* **57** 603–19.

[11] Taer, E., Deraman, M., Talib, I.A., Umar, A.A., Oyama, M. & Yunus, R. M. 2010. Physical, electrochemical and supercapacitive properties of activated carbon pellets from pre-carbonized rubber wood sawdust by CO2 activation. Current Applied Physics 10:1071-1075

[12] Dolah B N M, Deraman M, Othman M A R, Farma R, Taer E, Basri N H, Talib I A, Omar R and Nor N S M 2014 A method to produce binderless supercapacitor electrode monoliths from biomass carbon and carbon nanotubes *Mater. Res. Bull.* **60** 10–9.