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Submission date: 26-May-2023 12:30PM (UTC+0700)
Submission ID: 2102213482
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Word count: 2847
Character count: 14468

Journal of Physics: Conference Series

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To cite this article: D Junipuspita *et al* 2019 *J. Phys.: Conf. Ser.* **1191** 012020

Preparation of porous carbon made from candlenut shell (*Aleurites moluccana*) as a cathode for lithium ion capacitor

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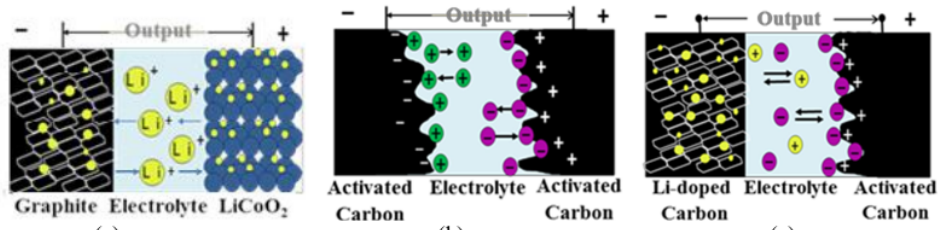
Abstract. Highly porous carbon from various biomass is known for its application as a cathode in energy storage devices. It has been receiving much attention for its ability to be the source of carbon precursor. This research focuses on the synthesis of activated carbon made from candlenut shell for the cathode of lithium ion capacitors (LIC). In the synthesis process, ultrasonic impregnation method was applied together with 3.5 M KOH and 3.5 M $ZnCl_2$ as the activators; processed at at 80°C for 2 hours with carbonization at 500 ° C and 650 ° C for 1.5 hours. The pore characteristics which are highly dependent on the activator can be determined by the Brunauer Emmett Teller (BET) method. The cathode was assembled from a mixture of activated carbon, Super P (carbon black) and polyvinylidene fluoride (PVDF) with the ratio of 85:5:10 (in wt%), respectively. The electrochemical properties show the highest capacitance of 717.587 mF g^{-1} with KOH as the activator. These results indicate that the activated carbon from candlenut shell would be potentially applied as precursor material for LIC cathode.

1. Introduction

Recently, fossil energy sources depletion and drastic increase of greenhouse gases effect have led the world in efforts to find solutions for such issues. One of the efforts is how to find more efficient renewable energy sources; in order to address the problem of increasing energy demand. Many of the renewable energy sources utilizations are directed to provide electrical energy which requires other energy storage systems such as battery and supercapacitor for further use [1]. In this case, lithium ion battery and supercapacitor are considered as promising candidates for energy storage system [2]. However, both candidates still show some limitations and need further improvement to increase their economical efficiency by using new material or optimizing their synthesis parameters [3].

Electrochemical energy storage system such as lithium ion battery (LIB) has high energy densities (ranging from 150 to 200 Wh kg^{-1}) and has storage mechanisms which are based on the oxidation-reduction (redox) reactions, but with low power density (<100 W kg^{-1}). On the contrary, supercapacitor has high power density (10 kW kg^{-1}) and high electrical charge which is stored in an electrical double layer (EDL), however, the energy density is low (5 - 10 Wh kg^{-1}). In this study, we



(a) (b) (c)

Figure 1. Basic configuration structures for (a) Lithium ion battery (b) Electrical double layer capacitor (EDLC), and (c) LIC [6, 7].

combined the storage mechanisms of LIB and supercapacitor to produce a new type of lithium ion capacitor (LIC) [2-5].

Figures 1 a, b and c show the structures of lithium ion battery, EDLC, and LIC. The structure of LIC is composed of two electrodes; the positive and negative electrodes consist of activated carbon and lithium ion, respectively. Positive electrode as a cathode here is to ensure high power density, while the negative electrode here is to provide high energy density. During the charging process, the anion absorption occurs on the surface of the positive electrode, whereas the hand lithium ion intercalation proceeds inside the bulk of the negative electrode. And during discharging, the anion absorption occurs on the surface of the positive electrode, while lithium ion deintercalation proceeds inside the bulk of the negative electrode. The process in the positive electrode was faster because it is non-faradaic, while in the negative electrode, due to the lithium ion exchange, the process was longer [6, 7].

The main factor that influence the performance of the supercapacitor is the electrode material [1], which usually used an activated carbon due to its large surface area [8]. Activated carbon produced from biomass is more preferred because its presence is abundant, cheap, easily obtained, and can be renewed [1]. Candlenut shell waste is one of the abundant sources of biomass. It has a hard texture because it contains high wood ingredients such as lignin, cellulose, and hemicellulose [9].

Basically, activated carbon can be produced by using two methods, namely, chemical activation and physical activation [10] to form a pore structure. For chemical activation, the activator is a key factor in forming the pore structure [8]. Therefore, a thorough study regarding the effect of specific activator on certain carbon source is still needed in order to understand the role of porosity and its relation on the LIC cell electrical properties. In this study, the activated carbon was made from candlenut shell by using KOH (potassium hydroxide) and $ZnCl_2$ (zinc chloride) as the activators. The main purpose of this study is to investigate the relationship between activators with capacitance and pore volume.

2. Experimental Methods

2.1. Carbonization of the candlenut shell

Candlenut shell was cut into small pieces with a length of 5 mm and then was cleaned by distilled water to remove dust and dirt. For activated carbon, 5 g of the sample was impregnated in the ultrasonic cleaner together with KOH 3.5 M and ZnCl2 3.5 M 20 ml at 80 °C for 2 hours. Then, the mixture was carbonized by the furnace for 90 minutes at 500°C and 650°C, with a rate increase of 10 °C per minute.

The obtained carbon was then crushed with a mortar and then washed with HCl 0.5 M to pH 2 and distilled water until the pH became neutral (pH 7). To separate the liquid and activated carbon, the centrifuge was used for 30 minutes at 3000 rpm. Furthermore, the activated carbon was dried in the oven for 24 hours at 100°C and then was sieved to 200 mesh.

2.2 Characterization of activated carbon

Activated carbon was characterized by BET to determine the surface area, total pore volume, pore distribution, and isotherm adsorption.

2.3 Coating proces

Activated carbon (85 wt%), Polyvinylidene Fluoride (PVDF) (10 wt%), and Super p (Carbon black) (5 wt%) and Dimethyl Acetamide (DMAC) solution (2.9 ml) were weighed in accordance with predetermined compositions. The materials were mixed with DMAC solution to obtain a homogeneous slurry form.

Then the slurry was printed on the aluminum foil with a thickness of 0.2 mm by doctor blade at a speed of 3.5 mm s^{-1}. The sheet was dried inside a dry box with a temperature of 70°C for 30 minutes. The cathode sheet design can be seen in Figure 2 (a). After it was dried, the cathode sheet was cut into a circle with a diameter of 1.6 cm as shown in Figure 2 (b).

2.4 Assembling the components of LIC supercapacitor

The LIC components were arranged in sequence; from the base, cathode, separator, LTO anode, and cover as shown in Figure 3. Then, a 65 µL lithium hexafluorophosphate ($LiPF_6$) electrolyte was being dripped on the separator. The process of punch to the cased supercapacitor was tightly closed.

2.5 Electrochemical Characterization

Electrochemical characterization of LIC was analyzed by Cyclic Voltammetry (CV) and by calculating the capacitance. The CV test was performed to confirm the presence of redox reaction.
The capacitance of LIC can be determined by the equation as follows [1]:

$$C = \frac{4 \times I \times t}{m \times \Delta V} \quad (1)$$

where C single i pecific capacitance (F g^{-1}); I is discharge current (mA); t is total discharge time (s); m is the mass of the electrode cell (g); ΔV is the potential difference during discharge (V).

(a) (b)

Figure 2. (a) Cathode sheet design and (b) Cell cathode design.

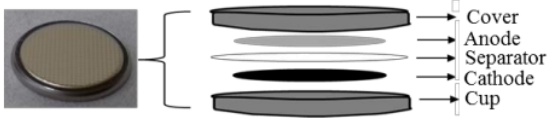


Figure 3. Components of LIC.

3. Results and Discussion

The effect of activator and temperature of carbonization on variations of pore size, pore volume, and surface area can be seen in table 1. With $ZnCl_2$ as the activator, the pore structure is amorphous, and the optimum temperature on the surface area and volume pores is 500°C, as formerly reported in the reference [10]. This happened because at high temperature (650°C), the increase in the ash content filled in the pore structure of activated carbon. Sample with KOH as the activator shows a larger surface area at 650°C because the impurities were easily released at higher temperatures as previously reported [5, 10].

Figure 4 shows the distribution of pores produced in the sample which is classified as micropore and mesoporous. According to IUPAC (International Union of Pure and Applied Chemical), pore size can be categorized into micropore (< 2 nm), mesopore (2 - 50 nm), and macropore (> 50 nm) [1]. N_2 gas was apllied to the activated carbon sample (BET process) to determine the size of the pore, volume, and surface area. In the process, N_2 gas were able to remove the impurities and prevented the

Table 1. Porosity data of candlenut shell activated carbon.

Sample	Activator	Carbonization Temperature (°C)	Pore size (nm)	Pore volume ($cm^3\ g^{-1}$)	S_{BET} ($m^2\ g^{-1}$)
KO2	KOH 3,5M	500	-	0.043	0
KO3		650	1.625	0.113	138.8
Zn2	$ZnCl_2$ 3,5M	500	3.780	0.08	43.86
Zn3		650	2.106	0.027	26.1

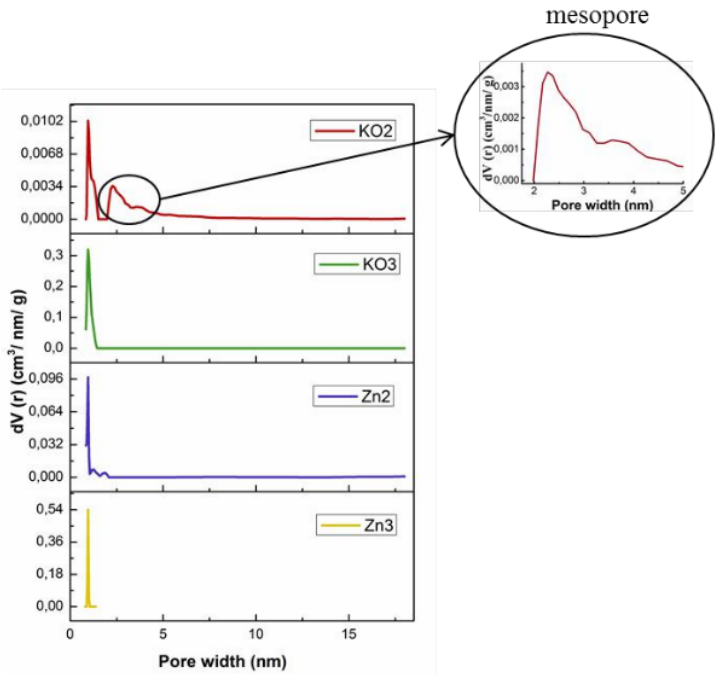


Figure 4. The pore distribution of candlenut shell activated carbon.

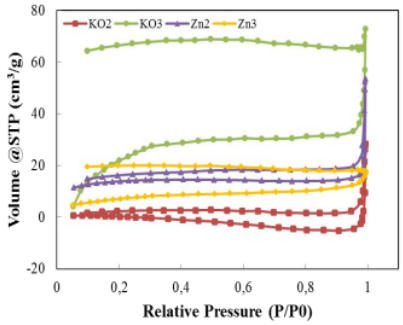


Figure 5. N_2 adsorption isotherm of candlenut shell activated carbon.

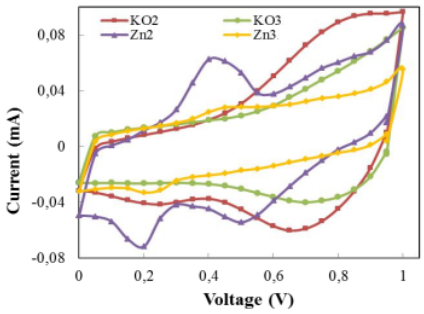


Figure 6. LIC cell cyclic voltammetry (CV) of candlenut shell activated carbon.

formation of tar on the surface of the pore. The amount of N_2 gas that was adsorbed by the activated carbon indicating the surface area and pore size. The zero BET value of KO2 sample is likely due to the pore complexity structure within the sample. This has made the N_2 adsorbate used for BET test was unable to access the deepest surface of the KO2 sample pore and can only access the outer surface of the sample. This has been confirmed in Figure 4, where KO2 sample posseses a considerable proportion of mesopores, than that of other samples. As a result, the overall adsorption isotherm curve created the negative BET gradient, giving a zero result when it was entered into an automatic BET calculation in the computer software.

In Figure 5, we analyzed the maximum adsorption volume of nitrogen gas absorbed in activated carbon. It has been shown that the relative pressure maximum is approximately 72.89 $cm^3\ g^{-1}$ at 650ºC for sample with KOH as the activator. The sample with high pore volume peak indicates that the absorption capacity of gas has also increased.

The CV hysteresis curve expresses a relationship between voltage (V) and resultant electric current (I), as shown in Figure 6. The presence of oxidation peaks are shown in the upper part of the curve. These peaks indicate the ion intercalation process into the cathode cell. In the bottom part, the curve shows the reduction peaks that clearly indicate that the absorbed ion subsequently undergoes deintercalation.

Deintercalation is a process of releasing ion or charge during cycle formation. The capacitance depends on the magnitude of the distance between the oxidation peak and the reduction peak. Based on the results, at 500°C, the sample with KOH as the activator has the highest capacitance. This is because the distance between reduction and oxidation peaks is the largest among all samples. With the increase of current distance in the CV hysteresis cycle, the value of the capacitance will also increase. Table 2 shows the calculation of specific capacitances of LIC cathode; with KO2 sample has the largest value.

Table 2. Specific capacitance cathode LIC of candlenut shell activated carbon.

Sample	m_k (g)	I (A)	t (s)	V (v)	C_{sp} (mF g^{-1})
KO2	0.0195	0.00000436	410	0.511	717.587
KO3	0.0231	0.00000348	410	0.511	483.493
Zn2	0.0200	0.00000309	405	0.499	501.583
Zn3	0.0200	0.00000383	405	0.499	621.703

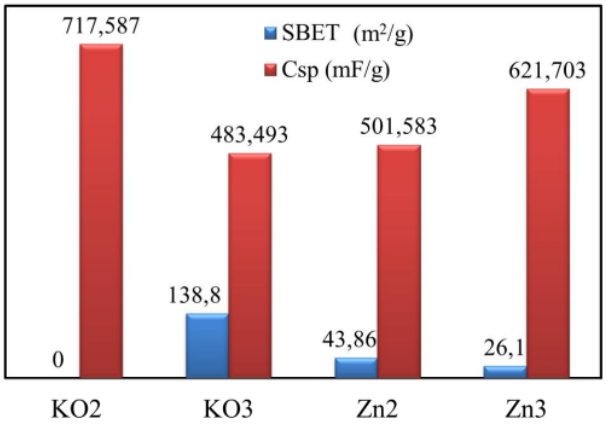


Figure 7. Relationship of Surface area S_{BET} (m^2 g^{-1}) and capacitance C_{sp} (mF g^{-1}).

Figure 7 shows the comparison between the activated carbon surface area and the LIC capacitance. KO2 has the highest capacitance of all samples. The high capacitance value is also affected by the relatively higher amount of mesoporous than the amount of micropore; as confirmed in Figure 4 for facilitating the occurrence of intercalation. Intercalation is a reversible process of insertion of ions into interlayer of an easily expanding structure (between silicone montmorillonite layers) without destroying the structure. The LIC capacitance value was also influenced by the activator; shown by the sample with KOH activator with the highest capacitance value of 717.587 mF g^{-1}.

4. Conclusions

LIC supercapacitor cathode was produced from activated carbon made from candlenut shell. The KO2 sample has the best capacitance value even though the pores size is too small and the pores were filled with impurities. The capacitance value is influenced by higher proportion of mesoporous, higher than that of micropore, hence facilitating the intercalation. In addition, activators have also affected the capacitance value. It is shown that the sample with KOH as the activator, has very good capacitance value. Additionally, KO2 sample was shown to reach high specific capacitance of 717.587 mF g^{-1}. This study shows that the activated carbon made from candlenut shell has high potential to be applied as one of the cathode precursor materials for LIC application.

Acknowledgments

We would like to show our highest gratitude and acknowledge the Research Center for Physics, Indonesian Institute of Sciences (LIPI) and all of the elements for their support in this research.

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