Longan Leaves biomass-derived renewable activated carbon materials for electrochemical energy storage

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Abstract. Biomass-based energy conversion and storage applications have proven to be the most effective technology for practical and sustainable applications. However, their further development was hindered by poor electrode performance. Naturally, abundant biomass is a green alternative carbon source with many desirable properties. This study presents a relatively easy approach for the synthesis of activated carbon-based electrode materials derived from natural biomass with an emphasis on supercapacitor applications. The selected biomass waste is Longan leaves. The precursor was converted into activated carbon through ZnCl\(_2\) impregnation at three different concentrations in high-temperature pyrolysis. All activated carbon confirmed a good amorphous structure. Furthermore, oxidative compounds were also found to have an effect on their electrochemical properties. Supercapacitor cells prepared in a two-electrode system exhibit high electro-capacitive properties with a specific capacitance of 169.83 F g\(^{-1}\) at a constant current density of 1.0 A g\(^{-1}\) in an aqueous electrolyte of 1 M H\(_2\)SO\(_4\). Furthermore, the optimum energy density was found in LF0.5 samples as high as 19.04 Wh kg\(^{-1}\) at a maximum power density of 124.37 W kg\(^{-1}\).

1. Introduction

Supercapacitors are an electrical energy storage technology that stores and releases energy by nanoscopic charge separation at the electrochemical interface between the electrode and the electrolyte [1,2]. Although the energy density of supercapacitors is very high compared to conventional capacitors, this energy density is still significantly lower than that of batteries [3,4]. Furthermore, supercapacitors with their performance are considered necessary to supply energy for a longer time period. This has prompted a strong interest in researchers to enhance the energy density of supercapacitors to get closer with batteries.

The electrode material is the main key component that determines the high performance of the supercapacitor [5,6]. Recently, porous carbon from waste biomass has become of great interest to researchers as a based material for electrodes for energy storage applications. This is due to the porous activated carbon from biomass waste offers fantastic material properties to support the electrochemical properties of supercapacitors, including a relatively very high surface area, providing a nanomaterial structure, a suitable pore combination, and relatively good chemical and thermal stability, willingness...
to abundant, renewable, and low cost [7,8]. Various reports have presented the potential of waste biomass to develop porous carbon materials as supercapacitor electrode applications [9–11].

Recently, Yang et al., 2019 reported a hierarchical porous carbon from mangosteen waste [12]. They transformed the mangosteen waste using carbonization and NaOH activation methods to modify porous carbon. The resulting surface area is 2623 m² g⁻¹ and an extraordinary specific capacitance of 357 F g⁻¹. Similar results were also shown by pitaya peel waste prepared using the KOH activation method with a surface area of 1870 m² g⁻¹ and a specific capacitance of 255 F g⁻¹ [13]. Furthermore, similar results were also found in several other biomasses such as Laminaria japonica [14], acacia leaves [15], coconut husk [16], watermelon [17], wood [18], etc. However, the porous carbon preparation described above still uses the addition of polymeric materials, metal oxide materials inhibiting further environmentally friendly applications of porous carbon-based materials.

Longan leaves is an agricultural plant that contributes to biomass waste. However, until now there has been no method of serious handling and high use value of them. The potential of Longan leaves waste is very large as activated carbon because it contains lignocellulose which is the basic material for high carbon content.

The objective of this research is to possess electrode material from porous activated carbon based on Longan leaf waste through a relatively environmentally friendly method using ZnCl₂ impregnation in high-temperature one-stage pyrolysis. For the record, the conversion of precursors into porous activated carbon does not use polymer materials, metal oxides, and adhesives. In addition, the chemical impregnation of ZnCl₂ was performed at three different concentrations including 0.3M, 0.5M, and 0.7M. In the optimum sample LF0.5, the electro-capacitive properties showed a high specific capacitance of 169.83 F g⁻¹ at a current density of 1.0 A g⁻¹ in an aqueous electrolyte of 1 M H₂SO₄. In addition, the optimum energy density was found in LF0.5 samples as high as 19.04 Wh kg⁻¹ at a maximum power density of 124.37 W kg⁻¹. Furthermore, the evaluation of electrochemical properties was tested through two methods including cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) which were effective and efficient in evaluating the performance of supercapacitors derived from porous carbon Longan leaves-based.

2. Materials and Methods

2.1. Materials

Longan leaves are obtained from the plantations of the Riau Province. Chemical activator agent ZnCl₂ and aqueous electrolyte H₂SO₄ were purchased by several brands such as Sigma Aldrich and Panreac Quimica Sau. DI water and distilled water are made in-house on a lab scale. Duck eggshell membrane was selected as an organic separator in supercapacitor cells rearrangement [19].

2.2. Preparation of Longan leaves activated carbon

Longan leaves were first cleaned with DI water and then randomly cut to ±2x3cm in size. Samples were sundried for two days followed by oven-dried for 24 hours, the mass loss samples were <6.0%. Dried Longan leaves were pre-carbonized and then crushed using a grinding machine to obtain Longan leaves powder with a particle size of ≤60μm. Furthermore, the powder samples were chemically activated using ZnCl₂ activator at different concentrations of 0.3M, 0.5M, and 0.7M. The chemically activated powder solution was oven-dried at 110 °C for 2 days. The activated powder was converted into a monolith without adhesive using a hydraulic press. Next, the monolith sample was pyrolyzed at high temperatures in an N₂ and CO₂ gas environment. The pyrolysis process consisted of carbonization from a temperature of 30 °C to 600 °C and physical activation from a temperature of 600 °C to 850 °C. To facilitate data analysis, the samples were labeled LF0.3, LF0.5, and LF0.7 for different concentrations of ZnCl₂. Finally, all monoliths were neutralized using distilled water.
2.3. Material Characterizations

The basic properties of monoliths were evaluated based on density loss through mass, thickness, and diameter measurements. Density is calculated using the standard equation and the volume is considered to be similar to tube space. Furthermore, the microcrystalline properties of activated carbon were reviewed using the X-ray diffraction (XRD) method at a 2θ angle ranging from 10°-60°. In addition, the microcrystalline dimensions and interlayer spacing were evaluated using the Debye-Sheerer equation and Bragg's law [20,21].

2.4. Electrochemical measurement

The electrochemical properties of the LF0.3, LF0.5, and LF0.7 electrodes were evaluated in a two-configurations supercapacitor cell. The supercapacitor cell is arranged coin-like stack consisted of two LF monolith electrodes, an organic separator, and an aqueous electrolyte of 1M H₂SO₄. Furthermore, the electrochemical properties were reviewed through cyclic voltammetry and galvanostatic charge-discharge methods with the CV-UR-Rad/Er 5841 and CD-UR Rad/Er 2018 instruments. These instruments were calibrated through the VersaStat II Princeton Applied Research with an error of approx. ± 6.05%. Specific capacitance, energy density, and power density are calculated by this method using their respective standard equations [22–25].

3. Result and Discussion

The chemical impregnation process at high-temperature pyrolysis significantly affects the dimensions of the monolith sample including mass, thickness, and diameter. The accumulation of this shrinkage was assessed through the density of the samples, as shown in Figure 1.

![Figure 1. Density shrinkage of LF0.3, LF0.5, and LF0.7 samples](image)

The density was evaluated in the process before and after high-temperature pyrolysis. Before pyrolysis, the density of monolith for samples LF0.3, LF0.5, and LF0.7 were 0.94, 0.96, and 0.88 g cm⁻³, respectively. After the chemical impregnation process at high-temperature pyrolysis, the monolith density possessed shrinkage of 34%, 44%, and 32%, respectively. Chemical impregnation at higher concentrations from 0.3M to 0.5M reduces monolith samples. This confirms that the chemical interaction of ZnCl₂ can evaporate volatile compounds, water content, and decompose lignocellulosic compounds [26,27]. Furthermore, the element Zn etches more carbon frameworks thereby increasing the formation of pores on the surface of the monolith sample. Moreover, ZnCl₂ acts as a dehydrating agent that can break the H and O chains in the functional groups of complex compounds in the
pyrolysis process, thereby reducing the sample density. Increasing ZnCl$_2$ concentration could hydrolyze ZnCl$_2$·nH$_2$O and produced oxychloride. Zinc oxide breaks down from oxychloride in a thermal process [28,29], thus LF0.5 sample had maximum density shrinkage of 44% of 0.53 g cm$^{-3}$. In addition, the higher chemical impregnation of sample LF0.7 slightly increased the density compared to LF0.5 by 0.59 g cm$^{-3}$. This is probably because too much chemical reaction can damage the carbon frameworks that have been formed thus the carbon pores collapse and it covers the pores below so that the density increases slightly. In addition, the byproduct ZnClO initiates the determination of the oxidizing compound in the sample. This was further confirmed through the XRD analysis discussed next. This result is almost the same as several previous studies [30,31].

The microcrystalline properties of Longan leaves-based activated carbon were further evaluated by X-ray diffraction (XRD). XRD pattern of LF0.3, LF0.5, and LF0.7 is shown in Figure 2. All of XRD pattern possessed two strong broadening peaks at 2θ angles of 23.103-24.321° and 42.497-43.534° (JCPDS No. 41-1487) which corresponds to 002 and 100 reflection planes speculated a hexagonal configuration of porous carbon and of graphite-like structure. These properties confirmed the turbostratic carbon behavior of activated carbon monolith longan leaves-based. This is similar with other study which studied previously such as sakura flower [32], banana leaves[33], wood [34], and acacia leaves [15]. Furthermore, the increasing ZnCl$_2$ impregnated were affected the broad peak, in case 002 reflection plane which detail in Table 1. The increasing ZnCl$_2$ impregnated from 0.3M to 0.5M could be move broad peak angel in 002 reflection plane to lower angle from 24.321 to 23.103 which speculated that existence of micropores and mesopores with random combination which improve the active site of ion diffusion and increasing the current density of the supercapacitor cell [35,36]. These properties may contribute to enhanced supercapacitor performance the LF0.5 sample which confirmed in CV and GCD analyses. Furthermore, several sharp peaks also reviewed in the XRD pattern, indicating the samples containing several crystalline-like compounds such as ZnO, SiO$_2$ and CaCO$_3$. The ZnO were found at 29°, 32° and 47°, CaCO$_3$ were observed at 43°, while SiO$_2$ was reviewed at 39° and 56° [27,37]. These compounds are contributed from the base components of Longan leaves biomass. In addition the ZnO compound may contribute from ZnCl$_2$ impregnated which decomposed in neutralized process.

![Figure 2. XRD pattern of LF0.3 and LF0.5 samples](image-url)
Table 1. The microcrystalline properties of LF0.3 and LF0.5 samples

| samples | $\theta_{002}$ (°) | $\theta_{100}$ (°) | $d_{002}$ (Å) | $d_{100}$ (Å) | $L_c$ (Å) | $L_s$ (Å) |
|---------|-----------------|-----------------|---------------|---------------|----------|----------|
| LF0.3   | 24.321          | 42.497          | 3.656761      | 2.125472      | 2.503821 | 2.857251 |
| LF0.5   | 23.103          | 43.534          | 3.846717      | 2.077215      | 6.194973 | 35.64316 |

The $d_{002}$ and $d_{100}$ interlayer spacing and $L_c$ and $L_s$ microcrystalline dimensions of LF0.3 and LF0.5 samples were calculated by using Bragg’s Law and Debye-Sheerer equation which detailed in Table 1. The $d_{002}$ and $d_{100}$ values are normal for porous activated carbon which similar with other reported. The $d_{002}$ increase of ±13.04% compared to the normal graphite. Moreover, $L_c$ value is associated with the predicted of specific surface area of the samples. In previous reported, $L_c$ is inversely proportional with high surface area. The relationship of $L_c$ with surface area of the activated electrode are given by empirical formula of SSA=$\frac{2}{\rho_x d_{002}}$ where $\rho_x$ is calculated by $d_{002}$ of graphite $\rho$ (graphite) and $d_{002}$ of sample $\rho$ (sample) are 0.33354 nm and 2.268 g cm$^{-3}$ [21,38]. In this empirical formula, the LF0.5 speculated has a high specific surface area compared with LF0.3 samples which could be enhanced the electrochemical supercapacitor performance.

Analysis of the electrochemical properties of activated carbon monoliths prepared from Longan leaves biomass is needed to optimize their electrochemical applications. Capacitive properties including specific capacitance, energy density, and power density were evaluated using cyclic voltammetry and galvanostatic charge-discharge techniques. Figure 3 shows the cyclic voltammetry (CV) curve of a sample of LF0.3, LF0.5, and LF0.7 in 1 M H$_2$SO$_4$ electrolyte.

![Figure 3](https://example.com/figure3.png)

**Figure 3.** The CV profile of LF0.3, LF0.5, and LF0.7 samples

All CV curves clearly show a distorted rectangular loop area and some curves show a slight spike in current. This shows that the capacitive performance is ideal for normal type electric double-layer electrochemical capacitors and is accompanied by pseudo-capacitance characteristics. Furthermore, the LF0.5 sample possessed a quadrilateral curve with increasing current density over a wide voltage range of 0.4-0.7 V confirming the faradaic pseudocapacitance associated with the presence of diverse functional groups or heteroatoms in the sample element distribution [39,40]. Several types of heteroatoms that contribute to this pseudo-capacitance include oxidative compounds which are confirmed in XRD pattern [41]. These elements are present in a small part, donated from the basic elements of biomass waste.

Moreover, this current spike at CV gradually weakens with an increase in the ZnCl$_2$ impregnated from 0.3 M to 0.7 M which is caused by the reduction of the heteroatom content especially oxygen element and it results in a decrease in the pseudocapacitance property. This analysis is in agreement...
with the density analysis previously presented, where the density decreases steadily with increasing ZnCl₂ impregnated.

**Figure 4.** The specific capacitance vs. scan rate of LF0.3, LF0.5, and LF0.7 samples

In addition, the rectangular closed area formed by the CV curve confirms the gravimetric specific capacitance properties, where the closed area of the CV curve of LF0.5 is the largest, followed by LF0.7, and LF0.3. Specific capacitance can be evaluated based on the CV curve through the standard equation that has been presented previously. Based on this equation, the specific capacitances are 96.98, 149.19, 126.27 F g⁻¹. ZnCl₂ impregnated from 0.3 M to 0.5 M can increase the capacitive behavior significantly from 96.98 F g⁻¹ to 149.19 F g⁻¹. This result is due to the changes in the material properties of the sample due to ZnCl₂ impregnation. Furthermore, further ZnCl₂ impregnated in the LF0.7 sample can decrease the specific capacitance from 149.19 F g⁻¹ to 126.27 F g⁻¹. The three- carbon monolith electrode LF0.3, LF0.5, and LF0.7 samples were also evaluated at different scanning rates of 1, 2, 5, and 10 mV s⁻¹. The resulting specific capacitance for applying the scan rate to each of these samples can be seen in Figure 4. Furthermore, it turns out that the scanning rate affects the specific capacitance of the carbon electrode, where increasing the scan rate higher from 1 mV s⁻¹ to 10 mV s⁻¹ reduces the specific capacitance of all samples. However, the three samples LF0.3, LF0.5, and LF0.7 still maintained a maximum of 42.20% of their initial specific capacitance at a current density of 10 mV s⁻¹.

**Figure 5.** The GCD profile of ACTCs supercapacitor cell
Furthermore, the galvanostatic charge-discharge (GCD) profile was evaluated to further confirm the electrochemical properties of the activated carbon electrodes. Figure 5 shows the galvanostatic charge-discharge (GCD) profile of LF0.3, LF0.5, and LF0.7 samples at a current density of 1.0 A g\(^{-1}\) with a scan rate of 2 mV s\(^{-1}\). The GCD profile shows an imperfectly symmetrical triangle shape with nonlinear characteristics which indicates the presence of a promising electrochemical double layer electrochemical capacitor in the sample and is followed by a faradaic reaction due to the distribution of sample elements containing heteroatoms indicating the presence of pseudo-capacitance in each sample [42–44]. This analysis is the same as the confirmed analysis of the CV curve in Figure 3.

![Figure 6. The Ragone of LF0.3, LF0.5, and LF0.7 samples](image)

**Table 2.** The electrochemical behavior of LF0.3, LF0.5, and LF0.7 samples

| Samples | \(C_{sp}\) (F g\(^{-1}\)) | \(E_{sp}\) (Wh Kg\(^{-1}\)) | \(P_{sp}\) (W kg\(^{-1}\)) | \(R\) (m\(\Omega\)) |
|---------|----------------|----------------|----------------|---------|
| LF0.3   | 107.02         | 14.45          | 104.36         | 7       |
| LF0.5   | 169.83         | 19.04          | 124.37         | 22      |
| LF0.7   | 137.67         | 16.95          | 105.62         | 18      |

Moreover, the potential for a sudden drop in the initial discharge profile looks different for each LF0.3, LF0.5, and LF0.7, due to the resistance to ions diffusion into the pores of the electrode material and the electrical conductivity of the carbon electrode [45]. The internal resistance values for each sample LF0.3, LF0.5, and LF0.7 are 7 x 10\(^{-3}\), 22 x 10\(^{-3}\), and 18 x 10\(^{-3}\) \(\Omega\), respectively. This resistance is seen to increase with increasing ZnCl\(_2\) impregnated from 0.3 M to 0.5 M. This is due to the presence of small pores that develop more and more at a relatively high temperature so that it slightly inhibits the accessibility of ions on the surface of the supercapacitor electrode. Based on the standard equation, the GCD profile shows the highest carbon electrode specific capacitance of 169.83 F g\(^{-1}\) for LF0.5, followed by LF0.7 and LF0.3 at 137.67 F g\(^{-1}\) and 107.02 F g\(^{-1}\), respectively. These results confirm the CV analysis previously presented in Figure 3. The energy density and power density generated by the GCD technique are also evaluated, as shown in Ragone plot, Figure 6. The optimum energy density was found in LF0.5 samples as high as 19.04 Wh kg\(^{-1}\) at a maximum power density of 124.37 W kg\(^{-1}\), as detailed shown in Table 2.
4. Conclusion
In summary, this study presents the potential of biomass waste as porous activated carbon monolith for electrode material of supercapacitor. ZnCl$_2$ impregnated in three different concentrations at high-temperature pyrolysis yielded highly porous activated carbon with amorphous structure confirmed. In symmetric supercapacitor cell, LF0.5 exhibited good electrocapacitive performance with highest specific capacitance of 169.83 F g$^{-1}$ at a constant current density of 1.0 A g$^{-1}$ in 1 M H$_2$SO$_4$ electrolyte solution. Moreover, LF0.5 samples displayed excellent capacitive retention, retain the specific capacitance of 42.20% of the initial capacitance in scan rate of 10 mV s$^{-1}$. These results promising good electrochemical performance of the sustainable Longan leaves biomass base material indicating that the precursor has great potential for supercapacitor applications.

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