Low-cost activated carbon bio-wasted-based for enhanced capacitive properties of symmetric supercapacitor

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Abstract. Low-cost, abundant, simple, and facile approaches to exhibit bio-waste-based porous carbon are needed to enhance the high performance of electrode materials as energy storage devices. Here, the bio-waste-based activated carbon with a low-cost and easy-to-prepare approach was studied via chemical impregnation of ZnCl₂ at single-stage integrated high-temperature pyrolysis. Furthermore, the activated carbon is prepared in the form of a monolith by optimizing the self-adhesive properties of the precursor. The application of different chemical concentrations can significantly improve the material properties with relatively good amorphous carbon structures. Furthermore, the maximum surface area was found to be 1129 m² g⁻¹. Moreover, it exhibits high electrochemical behavior with a specific capacitance of 145 F g⁻¹ at a constant current density of 1.0 A g⁻¹. In addition, the highest energy density was found at 16.25 Wh kg⁻¹ with a maximum power density of 82.70 W kg⁻¹ in 1 M H₂SO₄ aqueous electrolyte. These results confirm a low-cost and simple strategy for the synthesis of bio-waste-based activated carbon as an electrode material to enhance the capacitive properties of supercapacitors.

1. Introduction
Supercapacitor as known as electrochemical capacitor is a new type of energy storage device with wide application prospects and high economic value compared to other energy storage devices such as conventional capacitors and batteries. Supercapacitors, in particular the electrical double layer (EDLC) type have a much higher energy density than conventional capacitors and a much higher power density than batteries [1,2]. Energy is an important component of the world that supports the improvement of people's social life. Until now, fossil oil is still the main energy source even though their reserves are dwindling. This triggers the development of renewable energy and energy conversion systems, particularly in renewable energy from bio-waste. Bio-waste can come from various sources such as industrial waste, forest residue, water waste, and agricultural residues. The total supply of bio-waste worldwide is in range from 97-147 EJ/year of which about 38-45% comes from agricultural waste, followed by forest residues and industrial waste [3]. This increasing volume of bio-waste calls for economically viable countermeasures to reduce their environmental impact. Currently, bio-waste is widely used as a raw material to produce energy. However, because of its superiority, which is rich in lignocellulosic components, which are the main source of carbon, it has led to the use of bio-waste as a
precursor for activated carbon with wider applications [4]. Several potential applications of activated carbon that are attracting attention include absorption, water purification, removal of pollutants, solvent recovery, support catalyst, and energy storage. Energy storage devices such as batteries and supercapacitors are electrical devices that mostly use bio-waste-based activated carbon as their basic component. Apparently, in addition to saving costs and facilitating the fabrication process, the use of porous carbon bio-waste-based can also enhance electrochemical performance [5].

Facile techniques that are often used to convert bio-waste into activated carbon are physical activation and chemical activation. Physical activation involves gasification of N₂, Ar, CO₂, and H₂O at relatively high temperatures ranging from 600-950 °C [6]. On the other hand, chemical activation is performed by mixing a chemical solution with a bio-waste precursor, usually in powder form. Several chemical activating agents were reported such as KOH, K₂CO₃, NaOH, ZnCl₂, and H₃PO₄ which were pyrolyzed at high temperatures ranging from 400-800 °C [7,8]. Among the two, physical activation is considered the most frequently used technique because it is relatively easier to control. However, chemical activation is of particular concern because the yield of pure carbon is higher than physical activation. In addition, the single-stage chemical impregnation process produces material characteristics that are easily controlled according to their experimental conditions, particularly in the pore size distribution and high specific surface area. Several studies have reported these techniques to obtain activated carbon as electrode with outstanding material properties including high surface area, controlled pore distribution, nano-sized structure, high conductivity and excellent electrochemical properties [9,10]. Recently, Roy et al., 2021 converted banana leaves into porous carbon nanosheets through chemical activation of K₂CO₃ for supercapacitor electrode materials [11]. They have exhibited a high specific surface area of 1459 m² g⁻¹ with capacitive properties of 190 Fg⁻¹. Furthermore, Ji et al., 2021 have used KOH activation to fabricate porous carbon derived from garlic peels with an ultrahigh specific surface area of 3325.2 m² g⁻¹ [12]. They demonstrated an extraordinary specific capacitance of 424.42 F g⁻¹. Similar results were also shown for several bio-waste precursors such as mangosteen peel [13], pitaya peel [14], and pineapple leaves [15]. However, most of the reported bio-waste-based activated carbons still use adhesives such as PTFE and PVP to form solid carbon for supercapacitor electrodes. The synthetic adhesives could directly reduce and inhibit application performance further, thus it is necessary to study the potential identification of porous carbon based on bio-waste without the adhesives materials. One of the bio-waste that has the potential as activated carbon-based material is Terminalia catappa leaf. In addition to their abundant availability, renewability, and relatively no cost, these precursors have relatively high carbon fixed values of up to 74.49% with confirmed heteroatom presence [16]. Furthermore, their nanofiber-containing structure allows the electrode material to have high electrical conductivity properties [16,17]. These characteristics are needed to enhance the high performance of electrochemical energy storage devices.

In this study, bio-waste-based activated carbon was prepared through a low-cost and simple approach with chemical activation of ZnCl₂ in an integrated single-stage pyrolysis. Three different concentrations were applied to the precursor to optimize the porous properties of the carbon including 0.3M, 0.5M, and 0.7M. Furthermore, the activated carbon is designed in the form of a monolith by optimizing the self-adhesive behavior of the precursor. All samples confirmed good amorphous carbon properties with the highest specific surface area of 1182 m² g⁻¹. Moreover, the electrochemical properties of the electrode material were evaluated in the symmetric supercapacitor cell. The highest specific capacitance of 145 F g⁻¹ was found in the 0.5M ZnCl₂ impregnated sample with a maximum energy density of 16.25 Whkg⁻¹. These results confirm a low-cost and simple strategy for the synthesis of bio-waste-based activated carbon as an electrode material to enhance the capacitive properties of supercapacitors.
2. Materials and Methods

2.1. Preparation of activated carbon Terminalia cattapa leaf-based

The precursor of the biomass used for activated carbon is the dried *Terminalia cattapa* leaves which are found throughout the city of Pekanbaru. Conversion of the precursor into activated carbon begins with the drying and cutting process with a size of 1x2cm. The dried *Terminalia cattapa* leaf were converted into powder form through the process of crushing, grinding, and sifting to obtain a powder size of <60μm. Furthermore, 30g of powder sample was prepared and then mixed with ZnCl₂ solution at different concentrations including 0.3M, 0.5M, and 0.7M. This sample was then dried through a vacuum oven at 110°C for 48 hours. The ZnCl₂ impregnated powder was converted to monolith by optimizing the self-adhesive properties of the precursor through a hydraulic press instrument. A total of 15 samples were placed in a horizontal furnace tube for carbonization and physical activation in a one-step integrated system. Carbonization was performed in an N₂ gas environment with a maximum temperature of 600 °C, then physical activation was carried out in a CO₂ gas environment at a maximum temperature of 850 °C. Finally, it obtained *Terminalia cattapa* leaf-based activated carbon (ACTC) with variable concentrations of ZnCl₂ (0.3, 0.5, and 0.7M). The process of neutralizing monolith samples was possessed by immersing the samples in DI water until their pH was 7.0.

2.2. Material Characterizations

The material properties of ACTCs were evaluated through several reviews including changes in density, and microcrystalline structure. Changes in density were evaluated by measuring the mass, thickness, and diameter of the monolith sample. These measurements were repeated before and after the high-temperature pyrolysis. Density is calculated by the standard formula. Furthermore, the microcrystalline properties of the samples were assessed by means of X-ray diffraction technique with CuKα as the source. Interlayer spacing and microcrystalline dimensions were evaluated using Braggs law and Debye Sheerer equation, as shown in formulas 1, 2, and 3 [18]. In addition, through XRD the specific surface area can be predicted using empirical equations as previously reported, the detailed equations are shown in equation 4 [19,20].

\[
d_{002/100} = \frac{n\lambda}{2 \sin \theta_{002/100}}
\]

\[
L_c = \frac{0.89\lambda}{\beta \cos \theta_{002}}
\]

\[
L_a = \frac{1.94\lambda}{\beta \cos \theta_{100}}
\]

\[
SSA_{XRD} = \frac{2}{\rho_{XRD} L_c}
\]

Where

\[
\rho_{XRD} = \frac{d_{002\text{graphite}}}{d_{002\text{sample}}} \cdot \rho_{\text{graphite}}
\]

2.3. Electrochemical measurement

Electrochemical properties of ACTCs electrodes were evaluated in a symmetric supercapacitor system through cyclic voltammetry and galvanostatic charge-discharge methods. The supercapacitor cell is designed to resemble a coin layer consisting of two ACTC electrodes, an organic separator, and an aqueous electrolyte of 1 M H₂SO₄. The working mass of the electrode used is ±0.0120g. Specific capacitance was obtained from CV and GCD measurements via standard equations 6 and 7 [21,22]. In addition, different scan rates including 1, 2, 5, and 10 mV s⁻¹ were also applied to the CV technique.
Furthermore, energy density and power density were evaluated using equations 8 and 9, based on GCD measurements [23,24].

\[
C_{sp} = \frac{l_c - l_d}{s \cdot m}
\]

\[
C_{sp} = \frac{l \cdot \Delta t}{m \cdot \Delta V}
\]

\[
E_{sp} = \frac{l \cdot V \cdot \Delta t}{m}
\]

\[
P_{sp} = \frac{E_{sp}}{t}
\]

3. Result and Discussion

3.1. Materials behaviors analysis

Changes in the density of ACTCs before and after the pyrolysis process are shown in Figure 1. All chemically impregnated samples at different concentrations showed a decrease in density after the pyrolysis process. Before pyrolysis, samples ACTC0.3, ACTC0.5, and ACTC0.7 had densities of 0.9980, 0.9283, and 1.0743 g cm\(^{-3}\), respectively, with a mean standard deviation of ±0.066. The high-temperature pyrolysis carried out including carbonization and physical activation significantly affected all the dimensions of the sample monolith such as mass, thickness, and diameter [15,25]. Reduction of each of them degrades the density. After the pyrolysis process, the samples experienced a density degradation of 13.82, 33.92, and 28.73%, respectively. Their density values were 0.8500, 0.6134, 0.7656 g cm\(^{-3}\), respectively, with a mean standard deviation of ±0.045. Carbonization which acts as a precursor conversion process into pure carbon is the biggest contributor to reducing the density of the monolith sample. This process simultaneously completely evaporates the water content, volatile and other light compounds. Furthermore, at relatively high temperatures they begin to decompose lignocellulose complex compounds to produce highly pure carbon. However, the by-products they produce markedly inhibit the development of the carbon pore skeleton. Physical activation is required to remove ash and tar in the previous process so as to optimally produce a good pore framework. Moreover, ZnCl\(_2\) impregnation with higher concentrations from 0.3 M to 0.5 M can reduce the density of the monolith sample up to 33.92%. This is because more impregnation allows the reaction of carbon with ZnCl\(_2\) to be more intense so that it opens and forms relatively more pores through the evaporation of H\(_2\)O and CO\(_2\) [26,27]. This property benefits the precursor to exhibit high electrochemical properties in energy storage devices. However, the addition of more chemical impregnation to the monolith sample did not show the same data trend, as shown in the ACTC0.7 sample. This indicates that too much impregnation causes excessive expansion of the pore framework so that the carbon walls cannot maintain their shape. This initiates the collapse of the carbon framework and covers the empty space below it thus the sample density is slightly higher than the other two. Similar results were also found in previous studies [25,28].
The microcrystalline structure was evaluated through X-ray diffraction technique at 2θ angles of 10-60°, as shown in Figure 2. As confirmed by Figure 2, the XRD pattern for samples ACTC0.5 and ACTC0.7 displays at least two broad peaks at an angle 22-23° and 42-43° which are correlated with 002 and 100 reflection planes confirm good amorphous carbon properties based on biomass precursors [10,29]. Furthermore, the 100 reflection plane shows a smaller broad peak than the 002 indicating the presence of a graphite-like carbon structure [30]. Moreover, the ACTC0.5 and ACTC0.7 samples also perform several sharp peaks at different 2θ angles. This indicates that the sample also contains several small amounts of crystalline compounds such as SiO$_2$, CaCO$_3$, MgO, and ZnO [31,32]. The SiO$_2$ was found at 39° angles, CaCO$_3$ was confirmed at 32°, 33°, and 37° angles, MgO was indicated at 43° angles, and ZnO was found at 49° and 56° angles. The ACTC0.7 sample clearly showed more sharp peaks than the ACTC0.5 sample, as presented in Figure 2. This confirmed that more ZnCl$_2$ impregnation simultaneously produced more oxidative compounds. ZnCl$_2$ reacts with the carbon chain to produce the byproduct ZnO. This initiated the formation of other oxidative compounds in the ACTC0.7 sample. Although this oxidative compound inhibits the high performance of the electrode material, however, Zn and O elements can significantly provide a doping effect through a redox reaction resulting in a pseudo-capacitance effect on the electrode material [33,34]. This can improve the capacitive properties of supercapacitors, as further confirmed through electrochemical properties analysis.

Figure 1. Change in density of ACTCs

![Figure 1](image1.png)

Figure 2. XRD pattern of ACTC0.5 and ACTC0.7 samples

![Figure 2](image2.png)
Table 1. The interlayer spacing $d_{002}$ and $d_{100}$, microcrystalline $L_c$ and $L_a$ dimensions of ACTC0.5 and ACTC0.7 samples

| Samples  | $2\theta_{002}$ (°) | $2\theta_{100}$ (°) | $d_{002}$ (Å) | $d_{100}$ (Å) | $L_c$ (Å) | $L_a$ (Å) |
|----------|---------------------|---------------------|---------------|---------------|-----------|-----------|
| ACTC0.5  | 23,541              | 42,811              | 3,776         | 2,110         | 4,531     | 11,219    |
| ACTC0.7  | 23,057              | 43,86               | 3,854         | 2,062         | 17,664    | 6,390     |

Table 1 summarizes in detail the interlayers spacing $d_{002}$ and $d_{100}$, microcrystalline $L_c$ and $L_a$ dimensions, and SSA$_{XRD}$ specific surface area predictions for samples ACTC0.5 and ACTC0.7. The $d_{002}$ and $d_{100}$ show relatively normal values for amorphous carbon, as in previous studies [35,36]. Furthermore, the value of $d_{002}$ is considered to be 9.51% higher than that of graphite-structured carbon. This confirms the previous analysis that the obtained carbon is a structurally disturbed carbon towards high amorphous biomass-based. Microcrystalline dimensions are closely related to the prediction of pore properties and specific surface area of ACTCs activated carbon. As shown in equation 4, low microcrystalline dimensions indicate better porosity properties. Based on equation 4, the specific surface areas of the samples ACTC0.5 and ACTC0.7 are 1128 and 990 m$^2$ g$^{-1}$, respectively. This high specific surface area can improve the high energy density of supercapacitor. This was confirmed more clearly on CV and GCD analysis.

3.2. Electrochemical performance analysis

The electrochemical properties of activated carbon ACTCs were evaluated in a two-electrode system through cyclic voltammetry and galvanostatic charge-discharge methods. The CV profiles are shown in Figure 3 for three different electrodes of ACTCs including ACTC0.3, ACTC0.5, and ACTC0.7. At a scanning rate of 1 mV s$^{-1}$, all CV profiles exhibited a non-ideal rectangular shape confirming the normal electrochemical properties of the electric double layer of the biomass porous carbon-based material [37,38]. Interestingly, the ACTC0.7 sample performs a current spike in the voltage range 0.40 V to 0.85 V.

![Figure 3. The CV profile of ACTCs supercapacitor cell](image)

This is a pseudocapacitance effect due to the presence of heteroatoms especially oxygen. Oxidative compounds that were more involved in the ACTC0.7 sample as confirmed in the XRD pattern...
significantly gave a redox reaction effect and this could improve their capacitive properties. Furthermore, the closed-loop sweep area in CV profile confirms the capacitive value of the supercapacitor. ACTC0.5 sample shows the largest CV profile area confirming the highest specific capacitance followed by ACTC0.7 and ACTC0.3. Through equation 1, the specific capacitance for samples ACTC0.3, ACTC0.5, and ACTC0.7 are 78.36, 144.29, and 135.73 Fg⁻¹, respectively. The application of a ZnCl₂ concentration greater than 0.3M to 0.5M can significantly increase the specific capacitance almost twofold, as shown in the ACTC0.5 sample. This is because the ACTC0.5 sample has a high specific surface area of 1128 m² g⁻¹ allowing the sample to have more active contact sites for the electrolyte charge to diffuse on the electrode surface thus exhibit a maximum electrical double layer [39,40]. This result is considered higher than previous studies which used relatively complicated methods and relatively expensive based materials [41].

![Figure 4. The specific capacitance vs. scan rate of ACTCs supercapacitor cell](image)

Moreover, the three samples of ACTCs were also evaluated at different scan rates including 1, 2, 5, and 10 mV s⁻¹. This apparently affects the specific capacitance value, as shown in Figure 4. Even though the ACTC0.5 sample has the highest specific capacitance value at a scan rate of 1 mVs⁻¹, it retains only 30% of its specific capacitance at a scan rate of 10 mV s⁻¹. Likewise with the ACTC0.7 sample only maintains a specific capacitance of 34%. This is due to the uneven distribution of pore sizes so that the ionic charge transport path is blocked. This is a normal case for electrodes made from porous carbon-based biomass waste. Interestingly, the ACTC0.3 sample maintained the highest specific capacitance of 45% at a scan rate of 10 mV s⁻¹, although it did not have the highest specific capacitance at a scan rate of 1 mV s⁻¹.
Figure 5. The GCD profile of ACTCs supercapacitor cell

Galvanostatic charge-discharge measurements were performed to further confirm the electrochemical properties of the ACTCs electrode material. The GCD profiles for ACTC0.3, ACTC0.5, and ACTC0.7 are shown in Figure 5. All samples exhibited a distorted isosceles triangle shape confirming the relatively normal electrochemical properties of the electric double layer, particularly in the porous carbon-based electrodes of the bio-waste. Furthermore, the longer charge and discharge times of the GCD profiles indicate better electrochemical properties. ACTC0.5 showed the longest charge and discharge time with a specific capacitance of 145 F g\(^{-1}\), followed by samples ACTC0.7 and ACTC0.3 with capacitances of 122 and 67 F g\(^{-1}\), respectively. This result is relatively consistent with the CV analysis, as shown in Figure 4. In addition, the iR drop was not found significantly in all samples confirming the relatively low internal resistance of 16, 21, and 23 mΩ for samples ACTC0.3, ACTC0.5, and ACTC0.7. Moreover, energy density and power density were also evaluated using the GCD technique. As shown in Figure 6, Ragone plots for ACTCs electrodes confirm their energy density and power density. Sample ACTC0.5 showed the highest energy density of 16.25 Wh kg\(^{-1}\) at a maximum power density of 82.70 W kg\(^{-1}\), followed by samples ACTC0.7 and ACTC 0.3 of 13.76 and 5.97 Wh kg\(^{-1}\) at a power density of 72.83 and 65.53 W kg\(^{-1}\). This value is considered almost the same as other studies as shown in Table 2.

Figure 6. The Ragone of ACTCs supercapacitor cell
Table 2.

| Sources                                | \( \text{C}_{sp} \) (F g\(^{-1}\)) | \( \text{E}_{sp} \) (Wh kg\(^{-1}\)) | \( \text{P}_{sp} \) (W kg\(^{-1}\)) | Refs   |
|----------------------------------------|-------------------------------------|--------------------------------------|-------------------------------------|--------|
| Portunus trituberculatus Crab          | 128.5                               | 4.46                                 | 50                                  | [42]   |
| Pinewood                               | 349                                 |                                      |                                      | [43]   |
| Reeds                                  | 141                                 | 4.89                                 | 35.32                               | [44]   |
| Cotonier strobili                      | 346                                 | 33.04                                | 160                                 | [45]   |
| Frozen tofu                            | 170                                 | 72                                   | 889                                 | [46]   |
| Terminalia cattapa leaf                | 145                                 | 16.25                                | 82.70                               | This study |

4. Conclusion

Low-cost activated carbon derived from *Terminalia cattapa* leaf was successfully prepared by impregnation of ZnCl\(_2\) at high-temperature pyrolysis to enhanced electrochemical energy storage. Impregnation of ZnCl\(_2\) at higher concentrations significantly improved material properties with a specific surface area of 1128 m\(^2\) g\(^{-1}\). The electrochemical properties of the ACTCs were evaluated at a two-electrode system in a 1 M H\(_2\)SO\(_4\) electrolyte. ACTC0.5 sample exhibited the highest capacitive properties of 145 F g\(^{-1}\) with a maximum increase of up to 2 times. Furthermore, the highest energy density was found at 16.25 Whkg\(^{-1}\) at a maximum power density of 82.70 Wkg\(^{-1}\) in current density of 1.0 A g\(^{-1}\). These results confirm the high potential of a bio-waste-based activated carbon fabrication strategy as an electrode material for enhancing high-performance electrochemical energy storage devices of supercapacitors.

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