High Potential of *Averrhoa bilimbi* Leaf Waste as Porous Activated Carbon Source for Sustainable Electrode Material Supercapacitor

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Abstract. The energy cost-effective/free-environmental pollution concerns have an interest in bio-waste materials for the production of porous activated carbon, especially as electrode material for electrochemical energy storage devices such as li-ion batteries and supercapacitors. In this study, *Averrhoa bilimbi* leaf wastes were selected as a porous activated carbon source for sustainable electrode material supercapacitor. Porous activated carbons were prepared by chemical activation of 0.5 ml\(^{-1}\) sodium hydroxide solution at an optimum temperature of 800 \(^\circ\)C pyrolyze in an environment of N\(_2\) and CO\(_2\) gases. The monolith coin shape of activated carbon is maintained by optimizing the self-adhesive properties of the precursor without the addition of adhesive materials. All coin monoliths feature a turbostratic to highly amorphous carbon structure. Furthermore, the relatively high monolith dimensional shrinkage of 42.00\% initiated the development of a better pore framework carbon. In symmetric supercapacitors, electrochemical behavior confirmed a high specific capacitance of 149.04 F/g at a constant density of 1.0 A/g. Moreover, the maximum energy density was found of 10.50 Wh/kg at an optimum power density of 116.35 W/kg in an aqueous electrolyte of 1 ml\(^{-1}\) Na\(_2\)SO\(_4\). With bio-recycled waste, relatively easy preparation, and high electrochemical properties, porous activated carbon based on *Averrhoa bilimbi* leaf has great potential as a sustainable electrode material for supercapacitor energy storage applications.

1. Introduction

Wastes that accumulate valueless, especially biomass waste is a global problem in the world that causes environmental pollution, especially water-air pollution, and increased CO\(_2\) pollution. The potential for accumulation of global biomass waste is estimated to increase in the range of 97–147 EJ/year until 2030 [1]. The growth of this organic waste is influenced by the development of the latest technology in agriculture which is able to increase harvest yields up to several times that of conventional methods. However, their waste treatment is still far from applying high-level and sustainable methods. The largest potential supply of waste biomass comes from Asia, followed by Europe and North America. This high waste potential makes researchers focus on converting biomass into sustainable green energy [2]. Besides the growing demand for energy, biomass has its role to replace cost-effective renewable energy sources.
A high level of waste management has become a necessity considering that most cities are no longer able to control them properly. Stable city growth has drastically limited the availability of waste disposal land, so it is necessary to implement advanced waste management quickly and effectively, including considering waste technology into sustainable energy [3]. Waste to energy technology converts non-recyclable waste into usable forms of energy. The heat from burning the waste produces superheated steam in the boiler, and the steam drives a turbo generator to generate electricity [4]. However, their approach which instead requires complex techniques and complex equipment limits its wider application.

On the other hand, theoretical availability and cost modeling indicate that a large number of sustainable feed stocks can be provided for the production of sufficient porous carbon materials to meet the possible demand for high-performance-based materials in energy conversion systems and energy storage technologies [5,6]. The advantages of biomass-based carbon have demonstrated its achievements as a high-performance electrode material including high surface area, adjustable pore size distribution, presence of heteroatoms, high chemical and thermal stability, and good electrical conductivity [7-10]. The energy storage technologies that most often use biomass-based porous carbon as the base material for their electrodes are batteries and supercapacitors [11,12]. This study focuses on the contribution of porous carbon to the improvement of the performance of the supercapacitor device. Recently, the conversion of agricultural waste into porous carbon for electrode material has been of particular interest to materials science researchers [13-15]. This is due to their high potential in improving high-performance supercapacitors. For example, banana leaves have been converted to porous carbon through $\text{K}_2\text{CO}_3$ activation with a surface area of 1459 m$^2$/g [16]. It has confirmed the high capacitive properties of 190 F/g. Interestingly, they already have a unique combination of macro-, meso- and micropores that can increase the energy density to 59 Wh/kg. Furthermore, the natural porous carbon-based on Indicalamus leaves have produced a specific capacitance of 326 F/g with 3D hierarchical pore properties [17]. A similar potential is also found in other agricultural wastes such as neem leaves [18], waste tea leaves [19], garlic skin [20], and fallen ginkgo leaves [21]. However, some of them still require adhesives, besides the technique is considered relatively risky and toxic because the use of metal oxides and polymers limits their sustainability and environmental friendliness.

Here, a cost-effective/free-environmental pollution electrode material from porous activated carbon based on Averrhoa bilimbi leaf wastes was prepared for supercapacitor applications. Precursor powder was impregnated with sodium hydroxide on high-temperature pyrolysis. Furthermore, the carbon powder is converted into the form of a monolithic coin without the addition of adhesives materials. For the record, this study does not use polymer materials and metal oxides to maintain the basic properties of the precursor. In symmetric supercapacitors, electrochemical behavior confirmed a high specific capacitance of 257 F/g at a constant density of 1.0 A/g. Moreover, the maximum energy density was found of 25 Wh/kg at an optimum power density of 109 W/kg in an aqueous electrolyte of 1 M $\text{H}_2\text{SO}_4$. With bio-recycled waste, relatively easy preparation, and high electrochemical properties, porous activated carbon based on Averrhoa bilimbi leaf has great potential as a sustainable electrode material for supercapacitor energy storage applications.

2. Materials and methods

2.1. Synthesis of Averrhoa bilimbi leaf waste porous activated carbon

The Averrhoa bilimbi leaf waste collected from local farmers is cleaned, dried, and cut into pieces. To obtain the powder precursor, the sample was pre-carbonized at 250 °C and crushed through a grinder, and sieved with a size of 60 m. Furthermore, the precursor powder was chemically impregnated through a 0.5 ml$^{-1}$ sodium hydroxide solution on a hotplate at 300 rpm at 80 °C. The impregnated carbon samples were dried in a vacuum oven at 80 °C and further converted to monolith coins by maximizing the self-adhesive properties of the precursor material. A total of 15 samples of monolith coins were pyrolyzed at high temperatures, consisting of carbonization and physical activation. The carbonization was performed in an N$_2$ gas environment from a temperature of 30 °C to 600 °C, after
which it was continued with physical activation by flowing CO$_2$ gas to a maximum temperature of 800 °C. For comparison, the sample was pyrolyzed without a chemical impregnation stage. Moreover, the samples of the monolith coins were immersed in DI water until their pH was neutral technique (pH = 7). In detail, the conversion scheme of *Averrhoa bilimbi* leaf precursors into porous activated carbon is shown in Figure 1.

![Figure 1. The conversion scheme of *Averrhoa bilimbi* leaf precursors into porous activated carbon.](image)

2.2. Material characterizations

The *Averrhoa bilimbi* leaf-based porous activated carbon material behaviors were evaluated thoroughly through a review of monolith dimensional shrinkage and microcrystalline phase changes. The monolith dimensions include mass, thickness, diameter, and density. Their shrinkage value was evaluated against the high-temperature pyrolysis process. The density was evaluated based on Equation (1), where the volume of the monolith was calculated by Equation (2) [22].

$$\rho = \frac{m}{V} \quad (1)$$

$$V = \pi r^2 t \quad (2)$$

The $\rho$ is density of monolith carbon (g/cm$^3$), $m$ is mass of monolith carbon (g), $V$ is volume of monolith carbon (cm$^3$), $r$ is radius of monolith carbon (cm), and $t$ is thickness of monolith carbon (cm).

2.3. Electrochemical measurements

The working electrode was prepared from two *Averrhoa bilimbi* leaf-based carbon monoliths without adhesive materials with a mass loading of ± 6.1 mg bounded by an organic separator based on a duck eggshell membrane. Several aqueous electrolytes were selected to optimize the capacitive properties of the electrodes including H$_2$SO$_4$, Na$_2$SO$_4$, and KOH at a 1 M constant concentration. Electrochemical measurements were performed using two standard techniques such as cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD). CV was reviewed in a voltage window of 0.0 – 1.0 V at different scan rates such as 1, 2, 5, and 10 mV/s. The capacitive properties of the electrodes were evaluated according to the standard equation, as shown in Equation (3) [23,24],

$$C_{sp} = \frac{I_c - I_d}{s \times m} \quad (3)$$

where $C_{sp}$ is specific capacitance (F/g), $I_c$ is charge current (A), $I_d$ is discharge current (A), $s$ is scan rate (mV/s), and $m$ is mass loading (g). Furthermore, the GCD was assessed at a constant current density of 1.0 Ag$^{-1}$. The electrochemical properties were thoroughly evaluated including the specific capacitance, energy density, and power density of various different aqueous electrolytes. Specific capacitance, energy density, and power density are evaluated based on Equations (4), (5), and (6) [25,26],

$$C_{sp} = \frac{I_c - I_d}{s \times m} \quad (3)$$

$$E_d = \frac{1}{2} \int (C_{sp} - C_{sp0}) \, dV \quad (4)$$

$$P_d = \frac{E_d}{t} \quad (5)$$

$$P_d = \frac{E_d}{t} \quad (6)$$
where $C_{sp}$ is specific capacitance (F/g), $\Delta t$ is discharge time (s), $\Delta V$ is discharge voltage (V), $I$ is current (A), $E_{sp}$ is energy density (Wh/kg), and $P_{sp}$ is power density (W/kg).

3. Result and discussion

Porous activated carbon designed to a monolith coin-like with reduced use of adhesives material is prepared to maximize the materials high performance [27]. As we known, synthetic adhesive materials have insulating properties and it can increase the internal resistance and reduce the conductivity of the electrode-based material. This study applies a monolith coin shape to maintain the overall material properties to support high-performance supercapacitor electrodes. Monolith coins of porous activated carbon are prepared from scratch before the high-temperature pyrolysis. The pyrolysis process at high temperature performed in an integrated one-step includes carbonization and physical activation simultaneously reducing the dimensions of the monolith coin sample. These dimensions such as mass, thickness, diameter, volume, and density. The pyrolysis process that begins with carbonization from a temperature of 30 °C to a maximum temperature of 600 °C can significantly evaporate water, volatile and other light compounds in the form of H$_2$O and CO$_2$ [28].

![Figure 2. Change in density of porous activated carbon *Averrhoa bilimbi* leaf-based.](image)

Furthermore, this process also decomposes other complex compounds including hemicellulose, cellulose, and lignin at their respective temperatures. Hemicellulose is believed to evaporate completely at a temperature range of 150–240 °C, followed by cellulose at a relatively high temperature from 250 °C to 430 °C [29,30]. Lignin is a component of lignocellulose which has the longest decomposition temperature range from 150 °C to 700–800 °C [31,32]. All of this certainly reduces the dimensions of the monolith significantly, as shown in Table 1. Furthermore, because the carbonization process produces tar and ash by-products, a high-temperature physical activation process is required to maximize the desired change in material properties. Physical activation performed by
flowing CO_2 gas with a flow rate of 10 °C/min from a temperature of 600 °C to 800 °C can significantly remove tar and ash [9]. This process can also erode the carbon frameworks thus exhibit relatively more pores structures. Therefore, the combination of the carbonization and physical activation significantly reduced the mass, thickness, diameter, volume, and density of the monolith coin sample, as detailed in Table 1. Moreover, the chemical impregnated at the sample also showed almost the same effect. As shown in Figure 2, the density of the non-impregnated and KOH-impregnated samples decreased. However, their decline is considered much different, after the high-temperature pyrolysis process. The non-impregnated sample showed a density reduction from 0.97 g/cm^3 to 0.81 g/cm^3 at average deviation of ± 0.03, with a reduction percentage of 16.49%. The KOH-impregnated sample exhibited a density reduction from 1.00 g/cm^3 to 0.58 g/cm^3 at average deviation of ± 0.04, with a reduction percentage relatively higher as high as 42.00%. This is due to the chemical impregnated through 0.5 ml⁻¹ sodium hydroxide solution reacts with the precursor sample. Their reactions are detailed as follows [33,34].

\[ 6\text{KOH} + 2\text{C} \rightarrow 2\text{K} + 3\text{H}_2 + 2\text{K}_2\text{CO}_3 \]  
(7)

\[ \text{K}_2\text{CO}_3 \rightarrow \text{CO}_2 + \text{K}_2\text{O} \]  
(8)

\[ \text{K}_2\text{O} + \text{C} \rightarrow 2\text{K} + \text{CO} \]  
(9)

Sodium hydroxide etches the carbon and it exhibits the byproduct K_2CO_3 at relatively higher temperatures > 600 °C. Simultaneously, it also erodes the carbon walls by evaporating elemental H_2. Furthermore, an increase in the higher pyrolysis temperature causes K_2CO_3 to evaporate CO_2 and form K_2O. K_2O reacts with the carbon framework to form new pores by evaporating CO. The remaining K element is neutralized by immersing the sample in distilled water at the end of the synthesis process, as described in the previous subsection. Their overall reaction simultaneously reduces the density of the sample, as shown in Figure 2. As has been studied, this property is needed by the electrode-based material to improve the high performance of energy storage devices, especially supercapacitors. In comparison, the results obtained in this study are considered to be better than those of previous studies with different baseline precursors [35,36]. Of course, this also confirms higher electrochemical properties, as confirmed on CV and GCD analysis.

| Table 1. Dimensions of porous activated carbon at before and after high-temperature pyrolysis. |
|---|---|---|---|---|
| Dimensions | Non-impregnated Before pyrolysis | After pyrolysis | KOH-impregnated Before pyrolysis | After pyrolysis |
| Massa (g) | 0.68 | 0.27 | 0.71 | 0.12 |
| Diameter (cm) | 1.97 | 1.53 | 1.96 | 1.23 |
| Thickness (cm) | 0.23 | 0.19 | 0.25 | 0.17 |
| Volume (cm³) | 0.69 | 0.34 | 0.75 | 0.21 |
| Density (g/cm³) | 0.97 | 0.80 | 1.00 | 0.58 |

The electrochemical properties of porous activated carbon electrode material based on Averrhoa bilimbi leaf were evaluated optimally through cyclic voltammetry and galvanostatic charge-discharge techniques. The supercapacitor cells were prepared in a two-electrode symmetric system through coin layers consisting of Averrhoa bilimbi leaf-based carbon electrodes, separator, and electrolyte. The activated carbon monolith coins are designed with a thickness of 0.19 mm at a diameter of 8.00 mm. the average working mass of the electrodes ranges from 0.61–0.85 g. The separator was prepared by extracting the membrane on the duck eggshell. 1.0 ml⁻¹ sodium hydroxide solution was prepared as the aqueous electrolyte.
As shown in Figure 3, the CV profiles for both samples show a non-ideal rectangular shape indicating that the sample has an electrochemical double layer behavior which is relatively ideal for porous carbon-based materials from biomass waste. Furthermore, the CV curve does not imply any specific current spike across the voltage window 0.0–1.0 V. This confirms that the sample does not have pseudocapacitance due to the presence of heteroatoms. In addition, the high-temperature pyrolysis method has optimally removed the heteroatom properties of the precursor sample. At the relatively low voltage $U < 0.1$ V, the current density increased drastically in both samples indicating that the electrolytic ion charge diffuses on the sample surface rapidly and it fills the relatively smaller pores. An increase in the voltage window $0.2 V < U < 0.9$ V shows a relatively low increase in current density confirming that almost all the charge of the electrolyte ion has filled the small pores and they start the diffusion in the relatively larger pores.

Furthermore, a fairly sharp increase in the voltage window $0.9 V < U < 1.0$ V indicates the presence of macropores which are relatively confirmed to be clearly filled with charged electrolyte ions. This process automatically reverses direction when the voltages reach their maximum value of 1.0 V. This result actually forms a distorted rectangular curve, as shown in Figure 3. Moreover, the large area of the formed curve confirms the capacitive behavior of each sample. Based on Equation (3), the specific capacitances for the non-impregnated and KOH-impregnated samples are 13 F/g and 133 F/g. Interestingly, the combination of the KOH impregnation technique at high-temperature pyrolysis can drastically increase the capacitive properties of KOH-impregnated up to 10 times. This is due to the combination of chemical impregnation and physical activation can maximize the material properties of the precursors including their porosity, surface area, pore size distribution, and conductivity [37,38]. This is clearly seen through a review of the dimensional reduction of the monolith coins shown in Table 1 and Figure 2.

The KOH-impregnated sample had significantly the lowest density with a reduction of up to half which initiated more pore formation and increased the porosity of the based material. CV profiles were also evaluated at different scan rates including 1, 2, and 5 mV/s. As shown in Figure 4, the CV profile still displayed a distorted rectangular shape at a higher scanning rate of 5 mV/s, indicated that the sample has high electrochemical properties of the electrochemical double-layer. However, this increase in scan rate affects their capacitive properties, as shown in Figure 5. Both samples produce a smaller specific capacitance as the scan rate increases from 1 mV/s to 10 mV/s. This is influenced by the behavior of their pores which cannot be controlled thoroughly. This is considered to be common in biomass-based porous activated carbon. In addition, the KOH-impregnated samples were able to
maintain a specific capacitance of 72.82% indicating that their pores are relatively good for the based material of the electrode for the supercapacitor.

**Figure 4.** The CV profile of KOH-impregnated samples in different scan rate.

**Figure 5.** The specific capacitance vs. scan rate curve of porous carbon *Averrhoa bilimbi* leaf-based.
Furthermore, the electrochemical properties of the two samples were evaluated by means of a galvanostatic charge discharge at a current density of 1.0 A/g. As shown in Figure 6, both samples exhibited a non-ideal isosceles triangle shape confirming the good electrochemical properties of the electric double layer. In addition, the GCD profile did not indicate any pseudocapacitance effect in the two samples [39]. This analysis strengthens the CV analysis that has been presented previously. Moreover, the iR drop was not clearly found in the Non-impregnated samples whereas it was confirmed clearly in the KOH-impregnated samples indicating their internal resistance. Based on eq. (4), the Non-impregnated and KOH-impregnated samples have specific capacitances of 14.70 F/g and 149.04 F/g, respectively. This trend is similar to the results obtained from the previous CV analysis. Samples obtained by KOH impregnation at high-temperature pyrolysis have higher capacitive properties than samples that are only pyrolyzed without chemical impregnation. In this study, their capacitive increase was almost 10 times. However, KOH-impregnated samples had greater internal resistance than non-impregnated samples, as detailed in Table 2. This was due to the pore size distribution which could not be controlled by KOH impregnation. Furthermore, KOH impregnation is believed to produce predominantly small pores, thereby inhibiting the rate of diffusion of electrolytic ion charges on the electrode surface. In addition, energy density and power density were also evaluated through Equations (5) and (6). As shown in Figure 7, the Ragone plot confirmed the energy density and power density of the porous activated carbon electrode based on *Averrhoa bilimbi* leaf. The KOH-impregnated sample has a high energy density of 10.50 Wh/kg at an optimum power density of 116.35 W/kg. The results obtained were confirmed to be higher than supercapacitor electrodes with different precursors such as European deciduous trees [40] and bacterial cellulose [41].

**Figure 6.** The GCD profile of porous activated carbon *Averrhoa bilimbi* leaf-based.

| Samples          | \(C_{sp}\) (F/g) | \(E_{sp}\) (Wh/kg) | \(P_{sp}\) (W/kg) | R (mΩ) |
|------------------|------------------|---------------------|-------------------|--------|
| Non-impregnated  | 14.70            | 3.02                | 58.67             | 10     |
| KOH-impregnated  | 149.04           | 10.50               | 116.35            | 34     |

**Table 2.** The specific capacitance, energy density, power density, and internal resistance of porous activated carbon *Averrhoa bilimbi* leaf-based.
4. Conclusion
In summary, sustainable supercapacitor electrodes were obtained from activated carbon-based Averrhoa bilimbi leaf waste. KOH impregnation at high-temperature pyrolysis was chosen as a simple and cost-free strategy for the conversion of waste biomass precursors into porous activated carbon material. The based material is designed through a monolithic coin approach with no added adhesive materials. The basic material properties of carbon coins are evaluated by reducing the dimensions of their monoliths including mass, thickness, diameter, volume, and density. All dimensions of the monolith experienced a high reduction in the KOH-impregnated samples initiating the formation of good porosity properties. Furthermore, the electrochemical properties of the supercapacitor were evaluated in a two-electrode symmetric system. The confirmed maximum specific capacitance is 149.04 F/g at a constant current density of 1.0 A/g. In addition, the specific capacitance can be maintained at 72.08% at a scan rate of 10 mV/s. Moreover, the porous activated carbon obtained energy density is 10.50 Wh/kg at an optimum power density of 116.35 W/kg in an aqueous electrolyte of 1.0 ml⁻¹ Na₂SO₄. These results confirm the potential of Averrhoa bilimbi leaf as a porous activated carbon-based material for sustainable supercapacitor electrodes.

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