Study of the influence of different activator agents on the dimensions, mass, volume, and density of activated carbon monoliths for large-scale practical applications

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Abstract. By single step pyrolysis and different chemical activation (KOH and ZnCl₂) with different concentrations of 0.3 M, 0.5 M, 0.7 M, and 0.9 M, activated carbon monolith based on jengkol Pithecellobium jiringa shell wastes is obtained. The Samples are prepared in monolith form. The pyrolysis process begins with carbonization from room temperature to 600°C in the N₂ gas environment and followed by physical activation in the CO₂ gas environment at a high temperature of 850 °C for 2.5 hours. Monolith carbon is observed in the change of dimensions, mass, thickness, volume, and density. That it was observed that the pyrolysis process which includes carbonization and activation effectively reduces all the dimensions, mass and density parameters of carbon monoliths. Furthermore, the concentration of chemical activators dramatically exhibits excellent effects with increasing concentrations of activator agents, indicating that the porosity and highly micro-, and mesopores structure of activated carbon monoliths. This study is a model and reference for producing high-porous activated carbon from biomass waste for different practical applications.

1. Introduction

The conversion of biomass to biochar is one of the newest approaches for carbon sequestration in terrestrial ecosystems. In general, carbon materials can act as soil conditioners, promote plant growth, and improve soil physical and biological properties [1]. Another potential use of biochar is in fuel additives [2]. However, higher-value products could be obtained by converting biochar to activated carbon. Activated carbon (AC) has been widely used as a multipurpose adsorbent for gas separation, removal of organic pollutants, etc. due to its high surface area, large pore volume, and favourable pore size and good adsorption properties [3]. However, their high cost precludes their application in most practical applications. Of the various precursors, lignocellulosic biomass from agricultural and agro-industrial wastes is a promising feedstock, partly due to its abundant availability, renewability, and effective-cost production [4].

Recently, activated carbon from agricultural biomass waste such as wood [5], durian shell [6], coconut shells [7], mangosteen [8], cotton [9], rice husks [10], and others have been successfully prepared as an electrode material that promises high performance in energy storage devices, especially supercapacitors including EDLC and pseudocapacitors. Pithecellobium jiringa is one of the...
agricultural plants in Indonesia that belongs to the family of Fabaceae and classified as a subfamily of Mimosaceae which is usually called ‘jering’ or ‘jengkol’ [11] This agricultural plant is found in many Southeast Asia islands such as Sumatera, Kalimantan, Java, Malaysia, Thailand and Cambodia as they raw materials of typical traditional food [12]. In 2016, *Pithecellobium jiringa* production in Indonesia reached 57,404 tons/year and it is predicted to continue to increase [13]. *Pithecellobium jiringa* shell consists of high lignocellulose compounds of 56.84% and it is a potential as a source of activated carbon [14].

Basically, many reported that lignocellulosic wastes were converted to activated carbon through two steps [15,16], the first is a high-temperature pyrolysis process including carbonization and physical activation (300-900 °C), in which could be moisture, volatiles, and most of the non-carbon hetero elements in the biomass (oxygen, hydrogen, nitrogen, and sulfur). Chemical activation involves impregnating the raw material with chemical dehydrating agents including phosphoric acid, sulfuric acid, potassium hydroxide, and zinc chloride in an inert atmosphere [17–19]. However, this activated carbon is still in powder form which requires adhesives such as PVDF and PTFE to test the electrochemical properties of the supercapacitor. This adhesive is an insulator that inhibits the conductivity of the electrode supercapacitor material. Recognizing this drawback, several of our studies have been reported activated carbon from biomass waste in monolith/pellet form without the addition of adhesive agents [20–22]. These performed good physical properties including morphological structure, and pore development to enhance the electrochemical supercapacitor.

In this study, we focus on investigating the dimensions, mass, volume, and density of activated carbon monoliths for large-scale practical applications. Activated carbon was prepared from agricultural waste derived from *Pithecellobium jiringa* through single-step pyrolysis and KOH-ZnCl₂ activation. The concentration of KOH-ZnCl₂ was varied, such as 0.3, 0.5, 0.7, and 0.9 M. Reductions in mass, thickness, diameter, volume, and density were modelled to obtain accurate trend data. Furthermore, the structure morphology was also evaluated using the scanning electron microscopy method. Based on the results, this study is a model and reference for producing high-porous activated carbon from biomass waste for different practical applications.

### 2. Material and Methods

#### 2.1. Preparation of activated carbon monolith

Activated carbon monoliths were produced from *Pithecellobium jiringa* shell wastes through single step pyrolysis both carbonization and physical activation method. *Pithecellobium jiringa* shell is firstly cut into small pieces to facilitate the drying process. Furthermore, the samples were sundried for 3 days and carbonized in a vacuum oven at a temperature of 250 °C for 2.5 hours to obtain a green *Pithecellobium jiringa* shell that had self-adhesive properties. The next step involved smoothing using ball milling for 20 hours and sieving to obtain powder form with a particle size of 38-53 μm. The pre-carbonized powder is chemically activated using KOH and ZnCl₂ with varying concentrations of 0.3M, 0.5M, 0.7, and 0.9 M and non-chemical activation as a precursor. Chemical activation of pre-carbonized powder was carried out by hot-plate and magnetic stirrer at temperature of 80 °C with rotation rate of 300 rpm for 2 hours. The detail treatment was shown in Table 1.

| Sample Codes | Chemical Activation | Carbonization and physical activation |
|--------------|---------------------|---------------------------------------|
| None         | No Activation       | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| KOH3         | 0.3 M KOH           | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| KOH5         | 0.5 M KOH           | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| KOH7         | 0.7 M KOH           | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| KOH9         | 0.9 M KOH           | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| ZnCl3        | 0.3 M ZnCl₂         | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| ZnCl5        | 0.5 M ZnCl₂         | At 600°C in N₂ gas + at 850°C in CO₂ gas |
| ZnCl7        | 0.7 M ZnCl₂         | At 600°C in N₂ gas + at 850°C in CO₂ gas |
The activated powder was converted into monolithic carbon by using a hydraulic press with a pressure of 8 tons without any addition of adhesive materials. The monolithic carbon are pyrolyzed by single-step pyrolysis both carbonization and physical activation which start carbonized in the N₂ gas environment from room temperature to 600 °C with a temperature of resistance of 305 °C for 1 hour 38 minutes and followed by physical activation using CO₂ gas atmosphere to a high temperature of 850 °C for 2.5 hours in a gas flow rate of 1.5 liters/min and the temperature rate of 10 °C/min [23]. The monolithic carbon were washed to be a neutral (pH = 7).

2.2. Characterizations
The activated carbon monolith electrodes were characterized of density based on measurement of mass, diameters, and thickness. In addition, a polynomial function is used to modeling in this experiment for all reducing of dimensions, mass, diameters, thickness and density of activated carbon monolith. The equation of polynomial function can follow:

\[ y = a + bx + cx^2 \]  

(1)

The x variable is the condition of chemical concentration of chemical activation, y is dimensions, mass, diameters, thickness and density, a, b, and c are constant which obtain by modelling of experiment data. The morphology structures were reviewed by using scanning electron microscopy with JEOL-JSM 6510LA instrument at a magnification of 1000x in 15kV.

3. Result and Discussion
The reduction in mass, thickness, diameter, volume, and density were initial analysed that usually performed to evaluate the physical properties of activated carbon monoliths such as porous structure, surface morphology, and specific surface area. All measurements of monolith dimensions were carried out before and after the pyrolysis process. Mass is measured using an analytical balance while dimensions were measured using a digital caliper meter. The Volume is calculated using a standard formula with the assumption that carbon monolith is a tube while density is calculated using a standard equation based on mass and volume. In general, the pyrolysis process which consists of carbonization and physical activation significantly affects all dimensions of monoliths as well as chemical activation. The carbonization is to reduce the volatile content of the starting material to convert the resulting char with higher content of fixed carbon for activation purposes [24,25]. This process releases most of the non-carbon elements particularly hydrogen, oxygen, and nitrogen, in the form of gases and tars. The Physical activation is basically to develop further porosity and create some ordering of the structure which results in a highly porous solid of the activated carbon [26,27].

![Figure 1. Average reduction of mass using activator (a) KOH and (b) ZnCl₂](image-url)
However, chemical activation could dissolve the cellulosic components of the precursors and promote the formation of crosslinks [5,28]. The chemical reagent can decompose complex compounds in materials such as cellulose, hemicellulose, and lignin that produce nanomaterial structures in carbon monoliths such as nanofiber, nanosheet and nanosphere [29–31]. The results of this process may produce a maximum reduction of all dimensions indicating the formation of more pores and a high specific surface area on carbon monoliths. This advantage has the potential to have different practical applications such as absorbent, electrode material for supercapacitor energy storage.

Figure 1a and 1b show the average reduction of mass in carbon monolith based on an increase in the chemical activator. The reduction of mass significantly increases with increasing KOH and ZnCl₂ concentrations. However, these results have not shown a peak point indicating a maximum mass reduction to a concentration of 0.9M. Furthermore, the mass reduction in the KOH activator shows a more regular data trend compared to the ZnCl₂ activator. Due to the chemical activation using KOH to produce a high porosity and open up closed pores [32,33] while using ZnCl₂ developed numerous micropores so that it is a significant mass reduction in carbon monoliths [34,35]. The reduction of mass is also modeled using the polynomial function and constant have been obtained as in Table 2. This polynomial approach supports the experimental results to get a more precise maximum mass reduction.

| Table 2. Constanta of polynomial function for reduction of mass |
|---------------------------------------------------------------|
|                  | a   | b   | c   |
| KOH activator    | 54.58| 46.58| -23.60 |
| ZnCl₂ activator  | 53.64| 44.22| -26.42 |

Figure 2. Average reduction of thickness using activator (a) KOH and (b) ZnCl₂

The concentration of KOH and ZnCl₂ also affects the thickness of the carbon monolith as shown in Figures 2a and b. The increase in KOH concentration from 0 to 0.7M can increase thickness reduction regularly as shown in Figure 2a. Additional concentrations displayed different data trends. Based on this data it can be concluded that the maximum thickness reduction was found in the 0.7M KOH. However, this data trend does not match the modeling of polynomial functions. Meanwhile, the ZnCl₂ activator showed a significant increase of thickness reduction at a concentration of 0.3M and subsequently saturated to 0.5M. Further addition of ZnCl₂ concentration up to 0.7M turns out to have a far different data trend which shown in Figure 2b. A reduction in thickness decreasing at 0.7M is indicated that the pores expand in a larger direction so that the pore walls cannot maintain their shape.
and may collapse and reduce the carbon monolith thickness. The trend data on ZnCl$_2$ is fitted using the polynomial function and finds the maximum thickness reductions at a concentration of 0.35M ZnCl$_2$. Polynomial function modelling Constanta for activators KOH and ZnCl$_2$ are shown in Table 3. This is due to the chemical activation using KOH results in good pore development meanwhile chemical activation using ZnCl$_2$ pore development is so drastic and ZnCl$_2$ is able to destroy the sample [36] that it makes the sample brittle and the thickness plummeted.

**Table 3. Constanta of polynomial function for reduction of thickness**

|                | $a$  | $b$  | $c$   |
|----------------|------|------|-------|
| KOH activator  | 13.96| 25.51| -4.10 |
| ZnCl$_2$ activator | 13.91| 31.58| -44.62|

Figure 3 shows the reduction of diameter in carbon monolith after the chemical activation and the pyrolysis process based on differences in the concentration of KOH and ZnCl$_2$. The Increasing in chemical activation dramatically increases diameter reduction in carbon monoliths. This indicates that there are more developed pores. The KOH activator concentration showed an increase in mass reduction from 0 to 0.7M and otherwise, the addition of more concentration to 0.9M decreased the volume reduction. By using the polynomial approach, it could be seen that the maximum volume reduction is found at a concentration of 0.7M. Meanwhile, ZnCl$_2$ activation shows a slightly different trend. Increased volume reduction occurs at smaller concentrations compared to KOH. This data is also fitting using polynomial functions with Constanta shown in Table 4. Greater pore expansion as found in thickness reduction is also found in volume reduction, i.e. in 0.9M KOH and 0.7M ZnCl$_2$ concentrations resulting in decreased diameter reduction.

**Table 4. Constanta of polynomial function for reduction of diameter**

|                | $a$  | $b$  | $c$   |
|----------------|------|------|-------|
| KOH activator  | 19.96| 31.55| -24.72|
| ZnCl$_2$ activator | 20.87| 12.10| -10.42|

**Figure 3.** Average reduction of diameter using (a) KOH and (b) ZnCl$_2$.

The reduction of volume could be evaluated based on thickness and diameter data by assuming tubular carbon monoliths. In general, the data trends generated are almost the same as those shown by reduction of thickness and diameter. Reduction of volume increases with increasing concentrations of KOH and ZnCl$_2$ activators. However, the maximum reduction of the volume is found at different
concentrations. KOH activator showed a maximum volume reduction at a concentration of 0.7M while in the ZnCl₂ activator occurred at a lower concentration of 0.5M. This phenomenon indicates that activators KOH and ZnCl₂ affect the dimensions of carbon monolith. ZnCl₂ activator shows the development of more pores at smaller concentrations compared with KOH. In detail, Figure 4 showed a sample activated using KOH is greater than the average reduction of volume than the sample activated using ZnCl₂. It can be seen that the sample activated using KOH produces a high porosity compared to the sample activated using ZnCl₂. This is due to ZnCl₂ can destroy the samples.

Volume reduction data have been modelled using the polynomial function with constants shown in Table 5.

**Table 5. Constanta of polynomial function for reduction of volume**

|             | a   | b   | c   |
|-------------|-----|-----|-----|
| KOH activator | 46.03 | 47.29 | -28.48 |
| ZnCl₂ activator | 46.90 | 34.47 | -42.57 |

![Figure 4. Average reduction of volume using (a) KOH and (b) ZnCl₂](image)

Fig 5a and 5b show that the average reduction of density on each sample activated using KOH and ZnCl₂ with different concentrations. In the activated using KOH resulted in the greatest decrease in density at a concentration of 0.7M as well as the activation using ZnCl₂. The optimum concentration at a decrease in average density is at a concentration of 0.7M. activation using KOH produces a high porosity [38] this is due the samples are fragile during the carbonization and physical activation processes and on activation using ZnCl₂ produces a low porosity. This is because the ZnCl₂ activator is destructive to the sample [36].

**Table 6. Constanta of polynomial function for reduction of volume**

|             | a   | b   | c   |
|-------------|-----|-----|-----|
| KOH activator | 12.89 | 64.52 | -36.62 |
| ZnCl₂ activator | 12.89 | 64.52 | -38.62 |
Figure 5. Average reduction of density using (a) KOH and (b) ZnCl₂

Figure 6. Morphology of carbon electrodes from jengkol shell using KOH activation with magnification of 1,000 (a) none (b) KOH3 (c) KOH5 (d) KOH7 and (e) KOH9
Figure 6 shows the Scanning Electron Microscopy micrograph for all samples KOH activated at a magnification of 1,000. The all Figures represents none, KOH3, KOH5, KOH7, and KOH9 samples respectively. Figure 6a show the surface morphology with a more dense arrangement of carbon, irregular shape and the presence of small particles that cover the pores so that the carbon reduction of density to be a small. Figures 6b and 6e shows the surface morphology with a non-dense arrangement so that the pores are still visible and reduction of density to be a large. Figures 6c and 6d shows the surface morphology to produce nano fiber particles and reduction of density to be a very large. The nan fiber particles come from the addition of KOH concentrations and cellulose that exist naturally in the jengkol shell biomass [39].

Figure 7 shows the Scanning Electron Microscopy micrograph for all samples ZnCl2 activated at magnification of 1,000. The all Figures represented none, ZnCl3, ZnCl5, and ZnCl7 samples respectively. Figures 7a and 7b shows the surface morphology with a more dense arrangement of carbon and irregular shape so that the presence of small particles the cover the pores [23]. Figures 7c and 7d shows the surface morphology to produce nano sheet particles come from the addition of ZnCl2 concentrations. This is confirmed that different activator agent could to produce the different surface morphology structure.

Figure 7. Morphology of carbon electrodes from jengkol shell using ZnCl2 activation with magnification of 1,000 (a) none (b) ZnCl3 (c) ZnCl5 and (d) ZnCl7

4. Conclusion
This work demonstrates the reduction of dimensions, volume, and density of activated carbons with monolith/pellet form. The activated carbon monoliths are prepared from jengkol shell through by single-step pyrolysis and KOH-ZnCl2 chemical activation methods with the different concentrations of KOH-ZnCl2 impregnated. After pyrolysis and chemical impregnated, the dimensions, volume, and density reduced in the different concentrations of KOH-ZnCl2. Furthermore, the equation of a
polynomial function was ideal to use for initial evaluated the dimensions of carbon monolith based on enhanced concentration of chemical activation, especially KOH and ZnCl₂.

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