A study on activated carbon from Borassus flabellifer shell by chemical activation with potassium hydroxide for supercapacitor

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Abstract
The supercapacitor is significant in daily life. It is an alternative option that can storage energy. This research focused on studying the effect of Potassium hydroxide concentration content increasing surface area of the Borassus flabellifer shell charcoal (BFC), power density and energy density of activated carbon as electrode and cycles. Borassus flabellifer shell as agricultural waste was carbonized with tube furnace at nitrogen atmosphere by heated at 400 °C for 1 hour. Activated carbon with KOH (the mass ratio of KOH: BFC; 1:1, 2:1, 3:1, 4:1 and 5:1) (BAC) was heated at 900 °C for 2 hours. The results of the research show that suitable concentration of KOH: BFC to activated with higher pore volume and higher surface area. BAC-5 (5:1) is the highest pore volume of 1679.61 m²/g and the highest pore volume of 0.737 (cm³/g) but BAC-4 (4:1) is the best because it has a similar surface area and is economical, the highest pore volume of 1607.092 m²/g and the highest pore volume of 0.686 (cm³/g). BAC-1, BAC-2, BAC-3, BAC-4 and BAC-5 have average pore diameters, respectively, around of 1.609 nm., 1.641 nm., 1.742 nm., 1.708 nm. and 1.754 nm. The average pore size of products is microporous.

Keywords: Activated carbon, Borassus flabellifer shell, Electrode, Microporous, Potassium hydroxide

1. Introduction
Activated carbon or activated charcoal is a charcoal that must first be treated with a chemical or physical method. To cause the physical structure of the charcoal to create large numbers of microscopic pores or cracks at the nanometer scale. The pore size of the activated carbon can increase the efficiency of adsorption. When there are many microscopic pores, the absorption of the smaller molecules increases. Currently, researchers are studies on the use of carbon as a component of electrode for supercapacitor. Research into the porous of activated carbon is widely used (Dubey, R. and Guruviah, V., 2019). The Surface area of porous is important in terms of capacitance inside. If it has surface area and pore size at the mesopore or micropore, it has more electrons can be stored (Oyedotun, K.O., 2018).

However, carbon is a fundamental element of living things. Which can be obtained from plants (shell, flower, leaf, coir pith, etc.) and animals (Hu, L., 2016, Niu, L., 2019) but in various researches it has seen the human food resource as important (Chaweewan, 2019). Therefore, initiated the introduction of waste crops from agricultural crops used to make activated carbon from the study area or waste from agriculture come to take advantage. There is a research on one species of plant and not yet widespread, which has similar characteristics with coconut. It is palmyra palm or Borassus flabellifer. Raj used Borassus flabellifer flower (male flower) into activated carbon it has surface area 474.99 m²/g (Raj, F.R.M.S., Jaya, N.V., Boopathi, G., Kalpana, D. and Pandurangan, A., 2020). This plant is one kind of palm species. It is assumed to have originated in Africa. Later, it has been distributed in southern India.
and it spread to Southeast Asia including Thailand (Songkhla Provincial Agricultural Office, 1999). Most of the research uses all parts of the coconuts and some parts of palms that have similar species such as Lihong Yin used coconut fibers, it has surface area 2898 m²/g (Yin, L., 2016). Anjali Jayakumar used coconut leaf sheath -derived nitrogen doped carbon framework is developed and incorporated with nickel and cobalt metal (Jayakumar, A., Antony, R.P., Zhao, J. and Lee, J.M., 2018). Divyashree A. used coconut wastes, coconut fiber (Divyashree, A. et al., 2016). Activated carbon can be used as a heating fuel. Developed as a chemical adsorbent in water and supercapacitor. Many studies have been interested in using shell from this plant to make activated charcoal. Because of their high number of fibers and pores, they are well suited for the development for supercapacitor. Anukul K. Thakur used polypyrrole (PPY), Coconut shell activated carbon (AC) and Europium III oxide (Eu2O3). (Thakur, A.K., Choudhary, R.B., Majumder, M. and Gupta, G., 2017). Lin Peng mixed biomass wastes of coconut shell and sewage sludge, a high surface area is 3003 m²/g, a large pore volume 2.04 cm³/g (Peng, L et al., 2018). Kang Sun used coconut shell, a specific surface area is 1194.4 m²/g, total pore volume is 0.528 cm³/g, micropore percentage is 87.8 (Sun, K. et al., 2017) (etc. as shown in table 1).

Researchers then used the Borassus flabellifer shell (BF) in the research. In Thailand, it can be found a lot in many provinces, both the central and southern zones and it can also be found in many seasons. We using this resource because the price is very low. In addition to using the waste to be beneficial, it also helps reduce organic waste from the community. Another imperative thing is a substance that increases the porous surface area. There are 2 types of activation are physical reactivation (steam) (Misnon, I.I., 2015) and chemical activation (KOH, NaOH, ZnCl₂, K₂CO₃, H₂SO₄, etc.). Researchers use chemical activation because it can increase the porous surface area more than physical reactivation at same temperature. The optimum temperature is 600 - 900 °C (Sun, K. et al., 2017). Several studies have used strong bases to make it more micro porous. Potassium hydroxide is a very responsive substance. In this study, the effects of reaction conditions including Potassium hydroxide concentration content increasing surface area of the Borassus flabellifer shell charcoal (BFC).

Table 1 Comparison of activated carbons derived from various biowaste materials.

| Bio-waste precursor | Activation method | Mass ratio | Temperature (°C) | Surface area (m²/g) | Reference |
|---------------------|------------------|------------|------------------|---------------------|-----------|
| Pitch fiber         | NH3-gas          | -          | 900              | 658                 | (Torchala, K., 2015) |
| Brussel sprout      | KOH 2:1          | 800        | 2410             | (Li, J., 2016)      |
| Willow catkins      | KOH 1:1          | 600        | 645              | (Wang, K., 2015)    |
| Willow              | KOH 4:1          | 800        | 1775.7           | (Xie, L., 2016)     |
| Elm flower          | KOH 1:1          | 700        | 2048.57          | (Chen, H., 2018)    |
| Soybean             | KOH -            | 700        | 1749             | (Lin, G., 2018)     |
| Palm shell          | KOH 4:1          | 500        | 462.1            | (Misnon, I.I., 2015) |
| Palm shell          | steam            | 500        | 727              | (Misnon, I.I., 2015) |
| Sunflower stalk     | KOH 2:1          | 800        | 1505             | (Wang, X., 2018)    |
| Sakura              | KOH 4:1          | 750        | 1433.76          | (Ma, F., 2019)      |
| Borassus flabellifer flower (male flower) | KOH 0.3:1 | 600 | 474.99 | (Raj, F.R.M.S., 2019) |
| Coconut coir pith   | NaOH 3:1         | 700        | 2056             | (Sesuk, T., 2019)   |
| Coconut shell and sewage sludge | KOH 3:1 | 700 | 3003 | (Peng, L., 2018) |
| Coconut shell       | pyrolysis 1.75:1 | 900        | 1194.4           | (Sun, K., 2017)     |
| Pet-cokes and palm kernel shell. | K₂CO₃ | 680 | 318 | (Rashidi, N.A., 2020) |
| Coconut fibers      | KOH 4:1          | 850        | 2898             | (Yin, L., 2016)     |
2. Experimental section

2.1. Preparation of biomass precursor

*Borassus flabellifers* (BF) were obtained from the local area of Sathing Phra District, Songkhla Province in Thailand. The as-received BF were cleaned with water to remove soil and other natural contaminants and then dried with an oven at 105 °C for 24 hours to prevent mold. After that, BF were packaged in a zip-lock bag to prevent moisture.

2.2. Carbonization procedure

Approximately, 3 *Borassus flabellifers* per burning 1 time were used and then burned to charcoal. BF were carbonized by heating with a rate of 6 °C/min to 400 °C and maintaining this temperature for 1 h with calcined in a tubular furnace under a nitrogen atmosphere. After the *Borassus flabellifer* charcoal (BFC) cooled, BFC were crushed to a smaller size with a stone mortar. BFC powder was packed in a zip-lock bag to prevent moisture. Then put it in a box with silica desiccant.

The equation (1) used to calculate the yield of charcoal.

\[
\text{The yield of charcoal \( \% \text{wt} \) } = \frac{\text{Weight of Charcoal}}{\text{Weight of Raw material}} \times 100
\]

2.3. Activation procedure

Approximately 50.0 g of BFC was mixed with KOH 86.2%, AR grade (with a different mass ratio of KOH:BFC = 1:1, 2:1, 3:1, 4:1 or 5:1 in a beaker size 1,000 ml) product was stirred and immersed in beaker in closed box for 24 hrs. After slurry was filtered to leave only charcoal, it was activated by heating at a rate of 6 °C/min to 900 °C and maintaining this temperature for 2 h with calcined in a tubular furnace under a nitrogen atmosphere. followed by washing with dilute 0.1 MHCl solution and then deionized water until pH was adjusted to approximately 7. BAC was dried in oven at 105 °C for 24 hours. BAC was packed in a zip-lock bag to prevent moisture, then put in a box with desiccant silica. The final samples were denoted as BAC-x, where x represents the mass ratio of KOH to BFC.

The equation (2) used to calculate the yield of activated carbon.

\[
\text{The yield of activated carbon \( \% \text{wt} \) = } \frac{\text{Weight of Activated carbon}}{\text{Weight of Raw material}} \times 100
\]

2.4. Physical and chemical characterization

Static volumetric nitrogen adsorption isotherms were measured on a surface characterization analyzer (Micromeritics, ASAP 2460 version 2.01, USA) at 77 K. The specific surface area and porosity calculated by Brunauer-Emmete-Teller (BET) (surface area and porosity analyzer) technique. The morphology observation was carried out with Field Emission Scanning Electron Microscope (FESEM) images were used to examine the morphological properties of activated carbon by Merlin compact, ZEISS Microscopy, Jena, Germany from the Center for Scientific and Technological Equipment, Walailak University, Thailand. The total pore volume was determined at P/Po = 0.99. The micropore volume and pore size distribution were determined by the Harkins and Jura equation and non-local density functional theory (NLDFT). EDX mapping exhibits the presence of sulfur and oxygen in carbon matrix with variable wt.% with increasing carbonization temperature.
3. Results and discussion

3.1 Textural properties of activated carbons

The activated carbons adsorption isotherm graph is exhibited in Fig. 1. BAC-1, BAC-2, BAC-3, BAC-4 and BAC-5 isotherm curves present similar trends and shapes until reach a relative pressure (P/Po) of 0.99. All isotherm curves may be related to type I physisorption isotherm (according to the IUPAC classification). Type I isotherms are encountered with microporous powders whose pore size does not exceed a few adsorbate molecular diameters. Gas molecule, when inside pores of these small dimensions, encounters the overlapping potential from the pore walls which enhances the quantity adsorbed at low relative pressures. At higher pressures the pores are filled by adsorbed or condensed adsorbate, indicating little or no additional adsorption after the micropores have been filled. Physical adsorption that produces the type I isotherm indicates that the pores are microporous and that the exposed surface resides almost exclusively within the micropores, which once filled with adsorbate, leave little or no external surface for additional adsorption. This indicates that all samples have broader range of pore size distribution which includes a wider range of micropores (pores diameter less than 2 nm) and possibly with narrow mesopores (pores diameter between 2-50 nm) (Thommes, M. et al., 2015) and it in accordance with the pore size distribution of BAC-1, BAC-2, BAC-3, BAC-4 and BAC-5 have average pore diameter was in a range of 1.609 - 1.754 nm. as shown in Fig. 4.

![Fig. 1. Adsorption isotherm of activated carbons.](image)

![Fig. 2. Graph of the relationship of Concentration in the ratio of KOH: BFC of products to surface area and pore volume (Value obtained from Brunauer-Emmete-Teller (BET) technique as detailed in the table 2).](image)
The overall external and internal surface area of porous and porosity were calculated by Brunauer-Emmete-Teller (BET) technique. Fig. 2 and table 1 were shown properties of activated carbons at different mass ratio of KOH:BFC. The experimental are carbonized at 400 °C and varied from 1 to 5 of impregnation ratio at 900 °C of activation temperature. BET surface area of derived activated carbon was discovered in a range of 537.436 -1,679.609 m²/g and total pore volume was in a range of 0.216 - 0.737 cm³/g. Result was increasing impregnation ratio resulted in improvement of surface area and total pore volume, which has a similar trend as shown in Table 1. It was shown increasing the KOH concentration used for activation results in the surface area and pore volume increase as well. KOH is a stimulant that causes BAC to have an increased pore content and causing a microporous (less than 2 nm.). Although BAC-5 has the highest surface area (as shown in table 2) but the best sample is BAC-4 (KOH:C; 4:1, heat at 900 °C) because it has a similar surface area and it is economical, the highest pore volume of 1607.092 m²/g and the highest pore volume of 0.686 cm³/g.

3.3 Field Emission Scanning Electron Microscope analysis

The images obtained with FESEM can be used to provide a clear idea of the surface texture of porous materials. BAC-2, BAC-3, BAC-4 and BAC-5 depicted using FESEM at the magnification of 200 - 50000x. In Fig. 3 (a-d), it was magnified of 500x, BAC-2 was carbonized at 400 °C and mass ratio of KOH:BFC = 2:1, it was activated by heating at 900 °C. Fig. 3 (a) shows the nature of the pores of the BFC that are inherent in nature but there is corrosion caused by activation. Causing more holes in the outer surface. BAC-3, BAC-4 and BAC-5 were carbonized at 400 °C and mass ratio of KOH:BFC = 3:1, 4:1 and 5:1, there were activated by heating at 900 °C. Fig. 3 (b-d) shows a noticeably more porous surface, where the majority of porosities seen in the figure, micropore and radical pore size distribution as shown in Fig. 4. The results of FESEM were related to BET test, physical characteristics. Where more porosity tends to be in the same direction as the surface area and pore volume, as shown in Table 2.
Fig. 3. FESEM images of the samples; a. BAC-2, b. BAC-3, c. BAC-4 and d. BAC-5.

Fig. 4. The activated carbons pore size distributions
Table 3. Elemental composition of BAC-2, BAC-3, BAC-4 and BAC-5

| Sample   | C (%)   | O (%) | K (%) | Si (%) | Mg (%) |
|----------|---------|-------|-------|--------|--------|
| BAC-2    | 95.31   | 3.90  | 0.56  | 0.14   | 0.09   |
| BAC-3    | 97.15   | 1.95  | 0.62  | 0.29   | <0.01  |
| BAC-4    | 95.37   | 3.15  | 1.02  | 0.32   | 0.14   |
| BAC-5    | 86.06   | 8.64  | 4.83  | 0.13   | 0.33   |

Measured by EDS analysis.

3.4 Energy-dispersive X-ray spectroscopy

Element analysis data for BAC-2, BAC-3, BAC-4 and BAC-5 were present in table 3. The carbon content on BAC-5 (86.06%) was lower than BAC-2 (95.31%), BAC-3 (97.15%) and BAC-4 (95.37%) because it has mass ratio of KOH more than others. BAC-3 was the highest carbon element. Carbon was important, when it was used the electrode. Increased purity of carbon affects the adsorption capacity of an activated carbon, the higher the carbon purity the greater the adsorption power.
3.5 The yield of *Borassus flabellifer* charcoal and activated carbon

The Fig. 6 is the yield of *Borassus flabellifer* charcoal and activated carbon with 1:1, 2:1, 3:1, 4:1 and 5:1 impregnation ratio at different activation temperatures. The equation (1) for the yield of char and equation (2) for the yield of activated carbon. Yield of *Borassus flabellifer* charcoal was in a range of 27.58% - 28.52% by weight. Yield of activated carbons was in a range of 16.88% - 21.27% by weight. BAC-5 is the lowest yield of activated carbons. Result is increased mass ratio of KOH:C, it reduced yield of activated carbons (Nuilerd, T., 2018).

4. Conclusions

The *Borassus flabellifer* activated carbons (BAC) have qualified to use for electrode. BAC-1, BAC-2, BAC-3, BAC-4 and BAC-5 isotherm curves may be related to type I physisorption isotherm (according to the IUPAC classification). The best sample is BAC-4 (KOH:C; 4:1, heat at 900 °C) because it has a similar surface area and it is economical, the highest pore volume of 1607.092 m³/g and the highest pore volume of 0.686 cm³/g. Adsorption average pore diameter was in a range of 1.609 - 1.754 nm, respectively, as shown in Table 1. It was shown increasing the KOH concentration used for activation
results in the surface area and pore volume increase as well. KOH is a stimulant that causes BAC to have an increased pore content and causing a microporous (less than 2 nm.). Yield of Borassus flabellifer charcoal was in a range of 27.58% - 28.52% by weight. Yield of activated carbons was in a range of 16.88% - 21.27% by weight. The carbon content on BAC-5 (86.06%) was lower than BAC-2 (95.31%), BAC-3 (97.15%) and BAC-4 (95.37%) because it has mass ratio of KOH more than others. BAC-3 was the highest carbon element. In conclusion, BAC-4 should be used for electrode of supercapacitor and the microporous of Borassus flabellifer activated carbon can be developed by chemical activation with KOH.

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