The physical and electrochemical properties of activated carbon electrode made from pandanus tectorius

E Taer1, A Apriwandi1, Krisman1, Minarni1, R Taslim2, A Agustino1 and A Afrianda1

1Department of Physics, University of Riau, 28293 Simpang Baru, Riau, Indonesia
2Departement of Industrial Engineering, Islamic State University of Sultan Syarif Kasim, 28293 Simpang Baru, Riau, Indonesia

Email: erman_taer@yahoo.com; apriwandi95@gmail.com

Abstract. This research focused on analyzed the effects of carbonization and activation on the physical and electrochemical properties of carbon electrode made from pandanus tectorius. Carbon electrodes were varied in four different activation types such as non activated, chemical activation, physical activation, and chemical-physical activation. The samples were carbonized at temperature of 600 °C using N2 gas. Chemical activation was using 0.8 M KOH, and physical activation was done using CO2 gas at a temperature of 850 °C for 2.5 h. The density of the carbon electrode was analyzed by measuring mass and volumes. The morphology of the carbon electrode was reviewed by the Scanning Electron Microscope (SEM) method. Chemical element composition and Purity of carbon electrode was determined by Energy Dispersive X-ray (EDX). The degree of crystallinity was characterized by the X-ray Diffraction (XRD). The surface area of the carbon electrode can be evaluated based on XRD data. Electrochemical properties was evaluated using Cyclic Voltametry by testing two electrode mechanism in 1 M H2SO4 aqueous electrolyte. The activated carbon electrode based on pandanus tectorius with chemical-physical activation provides maximum surface area and maximum capacitance of 1144.82 m²g⁻¹ and 56 Fg⁻¹ respectively.

1. Introduction

Pandan (pandanus amaryllifolius) is one type of shrub, with pandanaceae family [1]. This type of family have 600 species of various sizes and shapes [2,3]. In the Indonesian forest, pandanus is often found as pandanus with thorns or pandanus tectorius. Pandanleaves is used for handicrafts such as made as mats and others [4]. In addition, the chemical composition of pandan leaves consists of 37.3% cellulose, 37.4% haem cellulose, 14.4% pentosans, 24% lignin and ash, and 2.5% extractive [5] so that pandan leaves become one of the potentially biomass material for activated carbon production at many application [5,6,7]. Biomass-based activated carbon show some physical properties such as amorphous structure, a high degree of porosity, an extended surface area, chemical stability and good conductivity [8]. The good physical properties of being a strong reason for the researchers to focus the research in electrochemical field as energy storage device component such as electrodes material in battery and supercapacitor [9]. Some of the activated carbon electrode from biomass used several preparation methods for supercapacitor applications, among others, Zhang et al. (2018) produces activated carbon from bamboo by KOH activation and high temperature of physical activation, resulting in high...
specific surface area of 2221.1 m² g⁻¹ and highest capacitance of 293 F g⁻¹ [10]. Hierarchically porous and heteroatom doped carbon derived from tobacco rods reported by Zhao et al. (2016) and it shows the highest specific surface area of 2898 m² g⁻¹ and specific capacitance of 266 F g⁻¹ [11]. Onget et al. (2012) produces activated carbon made from durian shell that was modified by the combination of ultrasonication and microwave irradiation techniques, the highest specific surface area as high as 648.64 m² g⁻¹ and highest electrode capacitance of 103.6 F g⁻¹ [12]. Pandan leaves has been used as activated carbon [5,6,7,13] but no one has reported it as a supercapacitor electrode. Preparation of activated carbon from the pandan leave raw material has been successful by using integrated carbonization and activation methods. The monolithic carbon electrodes prepared by variation of chemical and physical activation. Chemical activation is using 0.8 M KOH, whereas physical activation is performed by using CO₂ gas. Our results show that biomass-based activated carbon has a large surface area that provides ion transport between electrolyte and the carbon, resulting in good electrochemical properties. The results indicate that the activated carbons from pandanustectorius as electrode materials would be promising for supercapacitor applications.

2. Experimental Method
Pandanus monolithic activated carbon is produced by the preparation method previously reported [14]. Monolithic activated carbon was prepared in four different activations, such as KOH activation, CO₂ activation, combination of KOH-CO₂ activation and a sample without treatment as a basis. Based on activation variations, the samples were labeled as follows: AC/KOH, AC/CO₂, AC/KOH-CO₂ and AC/Untreatment. The KOH concentration is used 8 M. The pyrolysis process includes carbonization and physical activation carried out simultaneously in one step as previously reported [15]. Carbonization was carried out at a temperature of 600 °C using N₂ gas atmosphere with a flow rate of 1.5 L min⁻¹ followed by physical activation using CO₂ gas at a temperature of 850 °C for 2.5 hours with a flow rate of 0.5 L min⁻¹. The pyrolysis process for the AC/Untreatment and AC/KOH samples are only carbonized at a temperature of 600 °C. The capacitive properties of supercapacitor cells was elucidate by using two electrodes system. The arrangement of supercapacitor cell electrodes such as activated carbon electrodes, current collector, separator and electrolyte are arranged in sandwich form [8]. The current collector used is stainless steel 316-L type which produced by Goodfellow Cambridge Ltd., England. The separator is used as a duck eggshell membrane [16] while the electrolyte used is H₂SO₄ 1 M [17]. Characterization of the sample consists of physical and electrochemical properties. The physical properties of electrodes analyzed included density, degree of crystallinity, surface area, surface morphology and element content. Density is calculated by measuring the dimensions and mass of the carbon electrodes. The degree of crystallinity was characterized by using the XRD method with the X-Pert Powder Panalytical instrument with the Cu k-α light source and a wavelength of 1.5418 Å. The microcrystallite dimensions and interlayer spacing are calculated by using standard formulas [18,19] and bragg equation [20]. The surface area of the carbon electrode was calculated using the standard formula from microcrystalline height data obtained from XRD analysis [21]. The surface morphology was reviewed by using the SEM method with 5000 and 40000 magnifications. The element content was characterized by using the EDX method. The SEM-EDX characterization method uses the JEOL JSM 6510 LA. The electrochemical properties was measured by using the Cyclic Voltammetry (CV) method with the CV UR Rad-Er 5841 instrument and it calibrated with a 1280 solartron device. Specific capacitance is calculated using cyclicvoltammogram data with the formula [22, 23];

\[
C_{sp} = \frac{\Delta I}{\Delta m} \times s
\]

Where I = electric current, s = scan rate and m = mass of electrode.

3. Result and Discussion
3.1. Density analysis
The density of the activated carbon from pandanustectorius is shown in the Figure 1. Density is calculated by measuring the diameter, thickness and mass of carbon monolith (not shown here). The
sample density is presented with graphs before and after pyrolysis. The carbon sample without treatment has the highest density of 0.846 g cm$^{-3}$. As different activation treatments show the different density in the activated carbon sample. Samples with combination of KOH-CO$_2$ activation treatment had the lowest density of 0.685 g cm$^{-3}$, while a single activation sample such as AC/KOH or AC/CO$_2$ produces density in the range of untreated and combination KOH-CO$_2$ activation samples. The decrease in density is caused by the shrinkage mass and volume when chemical and physical activation process was performed. The activation process removal of elements other than carbon which causes the development of porous structures and increased carbon content. The mass decrease is due to the reaction of the carbon atoms with the activator agent while the volume decrease is related to the rearrangement of carbon atoms during the activation process [24] so the density was decreases.

![Graph showing density of carbon electrode from Pandanus tectorius](image1)

**Figure 1.** Density of carbon electrode from Pandanus tectorius

3.2. Degree of crystallinity analysis

![Graph showing X-ray Diffraction curve](image2)

**Figure 2.** X-Ray Diffraction curve

The XRD curves of carbon electrodes prepared by different activations are shown in Figure 2. All samples generally present the same curve. This curve is identical to amorphous carbon with two broadening peaks [25]. The result of X-ray diffractions are used to evaluate interlayer spacing and
The increasing of crystallinity means better conductivity. The increasing in baseline in the low angle area for activated carbon is probably caused by the residue from the pyrolysis process.

The sample structure parameters such as the interlayer spacings and microcrystallite dimensions are calculated and listed in Table 1. The microcrystallite height, in the range of 8.024-11.73 Å are almost identical to those of the activated carbon from other biomass materials, such as the durian shell, in the range of 10.58-36.21 Å [23]. These data are still in range of activated carbon. The different activation process give different effect on the interlayer spacing and microcrystallite dimension. The AC/KOH shows the smallest diffraction angle of 2θ and the AC/CO2 and AC/KOH-CO2 obtained the largest diffraction angle of 20. The diffraction angle 20 001 does not indicate a significant change in each sample treatment. Microcrystallite height produces varying data. The sample without treatment has the greatest Lc value. Along with the activation of KOH and CO2 given in the sample, the Microcrystallite height are decreased. The smallest Microcrystallite height is in the AC/KOH-CO2 sample. Activation of KOH-CO2 allows carbon electrodes to produce increased crystallinity due to the addition of KOH-CO2 at higher temperatures. The increasing of crystallinity means better conductivity. The increasing in baseline in the low angle area for activated carbon is probably to originate from the presence of micropores that are rich in carbon framework.

| Sample codes       | 2θ(002)(°) | 2θ(100)(°) | d(002)(Å)  | d(100)(Å)  | Lc(Å)   | La(Å)   |
|--------------------|------------|------------|------------|------------|---------|---------|
| AC/Untreatment     | 24.936     | 45.214     | 3.56794    | 2.00386    | 11.7256 | 20.3456 |
| AC/KOH             | 24.443     | 45.852     | 3.63878    | 1.97745    | 10.2181 | 22.4134 |
| AC/CO2             | 25.161     | 44.968     | 3.53655    | 2.01425    | 9.6355  | 26.5307 |
| AC/KOH-CO2         | 25.603     | 44.929     | 3.47649    | 2.01591    | 8.02419 | 10.2419 |

The microcrystallite height can be used to determine the specific surface area (SSA) of the electrode samples using empirical formula and it is shown in the Table 2. Based on the Table 2, The microcrystallite height is strongly associated with the surface area. A small microcrystallite height is required to produce a high specific surface area. Samples without treatment has the smallest specific surface area of 804.26 m²g⁻¹. The addition of KOH allows the development of good carbon pores so that the specific surface area increases to 942 m²g⁻¹. The CO2 Activation at a temperature of 850 °C indicates the presence of rich micropores in a carbon framework and produces a specific surface area of 970 m²g⁻¹. The combination of KOH-CO2 activation produces the highest specific surface area of 1145 m²g⁻¹. The addition KOH and CO2 activation developed micropores and more carbon pore so the carbon sample shown the highest specific surface area.

| Sample Codes       | Lc(Å)   | r(xrd)(gcm⁻³) | SSA(m²g⁻¹) |
|--------------------|---------|---------------|------------|
| AC/Untreatment     | 11.7256 | 2.1202        | 804.26     |
| AC/KOH             | 10.2181 | 2.0789        | 941.51     |
| AC/CO2             | 9.6355  | 2.1390        | 970.39     |
| AC/KOH-CO2         | 8.0242  | 2.1759        | 1144.82    |

3.3. Surface morphology analysis
The effect of activations on the surface morphology of activated carbon electrodes from pandanus tectorius is reviewed in Figure 3. The SEM characterization for all electrode samples using
magnifications of 40000x. Figure 3.a shown the sample without treatment presents agglomeration and larger particle size compare the other sample. The size of particle for AC/Untreatment is in the range of 0.685-0.153 μm. The AC/KOH sample displays a smoother surface morphology and a visible pore between particles and shown in Figure 3.b. The presence of pores between particles are indicated by a dark color. The particle size becomes smaller with a size range of 0.225-0.113 μm. KOH activation successfully reduces particle size because of the activation agent breaks the bonds between particles [26]. The Figure 3.c shown the CO\(_2\) activation sample, this activation produces particle sizes in the range of 0.442-0.202 μm. Combination of KOH-CO\(_2\) activation shows the smallest particle size morphology of 0.156-0.070 μm which shown in Figure 3.d. Combination of activation allows the reduction of the most particle size so as effect to the development of better pores for carbon electrodes from pandan leaves.

![Figure 3](image)

**Figure 3.** SEM micrographswith a magnification of 40000 times for a) AC/Untreatment; b) AC/KOH; c) AC/CO\(_2\); d) AC/KOH-CO\(_2\)

### 3.4. Chemical content analysis

The EDX spectrum presents element content of the electrode is shown in the Figure 4. The EDX analysis showed the level of element content [such as carbon and other elements] present in the electrode samples. This spectrum shows that the samples are composed of carbon, oxygen, magnesium, potassium and calcium. The highest peak was recorded for the carbon element. This high carbon peak indicates that carbon is the highest elemental content of the electrode sample which shown in percentage of 92.96%, 94.54%, 94.44% and 94.93% for AC/Untreatment, AC/KOH, AC/CO\(_2\) and AC/KOH-CO\(_2\), respectively. Chemical activation using KOH effects the quantity of oxygen, potassium and chlorine [26] so that the percentage of carbon changes from 92.96% to 94.54%. The CO\(_2\) activation at high temperatures also effects carbon elements. The combination of KOH-CO\(_2\) activation produces the highest carbon content so that a combination of activation indicated can erode and remove the other element content than carbon to the maximum for activated carbon from pandan leaves. The oxygen content is due to the presence of carbon and oxygen bonds at the
The activation process. The other elements such as potassium and calcium are the basic components of pandan leaves. Percentage composition of each element in the activated carbon electrodes is shown in Table 3.

| Element contents | AC/Untreatment | AC/KOH | AC/CO₂ | AC/KOH-CO₂ |
|------------------|----------------|--------|---------|------------|
|                  | Atom (%)       | Atom (%)| Atom (%)| Atom (%)   |
| Carbon           | 92.96          | 94.54  | 94.44   | 94.93      |
| Oxygen           | 5.54           | 4.79   | 4.96    | 3.50       |
| Magnesium        | 0.16           | 0.09   | 0.11    | -          |
| Potassium        | 0.22           | 0.21   | 0.10    | -          |
| Calcium          | 1.12           | 0.38   | 0.56    | 1.57       |
| **Totals**       | **100%**       |        |         |            |

Figure 4. The EDX spectra of the all samples

3.5. *The capacitive electrode analysis*

The cyclic voltammetry measurements are commonly used to test the EDLC cell performances that use an activated carbon electrode. The cyclic voltammograms of all samples in 1 M H₂SO₄ aqueous electrolyte at a voltage of 0-0.5 V with a 1 mV/s scan rate. The I-V curve formed a rectangular shape for carbon electrode material [27] which are shown in Figure 5. This shape type are represents the specific capacitance produced by pure carbon electrode [28]. The rectangular shapes of the CV curves imply a quick ion diffusion and good charge propagation in all electrodes at a lower scan rate. Based on Figure 5, the specific capacitance result of 24 F g⁻¹, 26 F g⁻¹, 43 F g⁻¹ and 56 F g⁻¹ for AC/untreatment, AC/KOH, AC/CO₂ and AC/KOH-CO₂ samples, respectively. The activations increase the electrode capacitive properties (from 24 F g⁻¹ to 56 F g⁻¹). KOH activation produces new pores and increases the surface area. The large surface area provides a large medium for the ions diffusion into the carbon matrix sample [26] so specific capacitance increase from 24 F g⁻¹ to 26 F g⁻¹ for AC/KOH sample. Activation using CO₂ in longer time tend to produce samples with dominant micropores [15] and it
can be result higher specific capacitance of 43 F g\(^{-1}\). In this study, combination of KOH-CO\(_2\) activation shown the highest specific capacitance of 56 F g\(^{-1}\), it means KOH-CO\(_2\) activation resulted the good combination pore and higher specific surface area so the specific capacitance reach maximum for carbon electrode based on pandanustectorius.

![CV curve for all samples](image)

**Figure 5.** The CV curve for all samples

4. Conclusion
The analyzed of physical and electrochemical properties of activated carbon electrode made from pandanus tectorius has successfully demonstrated. Activated carbon electrode prepared in four different activations such as KOH and CO\(_2\) activation by using one step pyrolysis process simultaneously. The addition of activator agents such as KOH or CO\(_2\) affects the physical and electrochemical properties of electrodes. Activator agents change the physical properties of the sample and improve the electrode capacitance properties. The carbon electrode has excellent physical and electrochemical properties for supercapacitor applications. The combination of KOH-CO\(_2\) activation is shown in the best physical and electrochemical properties compared with the other activation samples. The lowest density of resulting sampel is 0.685 g cm\(^{-3}\). Specific surface area produced reached of 1144.82 m\(^2\)g\(^{-1}\) with the highest carbon content of 94.93%. The superiority of physical properties supports good electrochemical properties with highest specific capacitance value of 56 F g\(^{-1}\).

Acknowledgements
The author would like to thank the Riau University through DIPA LPPM with the title "Carbon Nanofiber Electrodes Based Naturally Materials for Energy Storage Devices” with contract number: 661/UN.19.5.1.3/PP/2018. The author also thanks the SEM FMIPA ITB Laboratory, which has assisted in obtaining the SEM and EDX data.

References
[1] Mogejo J P 1982 *LBN-LIPI Nogor Suaka Alam* 16 21
[2] Kirtikar K R, Basu B D and Blatter E 1991 Indian Medicinal Plants Indian *Book Center* New Delhi, India 115
[3] Gurmeet S and Amrita P 2015 *J. Pharmacognosy and Phytochemistry* 508
[4] Stone BC 1999 Plant Resources of South-East Asia *Pandanus Edible Fruits and Nut Prosea, Bogor, Indonesia* 13 240
[5] Sheltami R M, Abdullah I, Ahmad I, Dufresne A and Kargarzadeh H 2012 *Carbohydr. Polym.* 88772
[6] VigneshwaranGV, JenishI and SivasubramanianR 2014 Adv. Mat. Research 984-985253
[7] Ismail M N, Aziz H A, Ahmad M A and Kamaruddin M A 2013 Int. J. Scientific Research in Knowledge 1388
[8] González A, Goikolea E, Barrena J A and Mysyk R 2016 Renew. Sustain. Energy Rev. 581189
[9] Inagaki M, Konno H and Tanaike O 2010 J. Power Sources 1957880
[10] Zhang G, Chen Y, Chen Y and Guo H 2018 Mat. Research Bulletin 102391
[11] Zhao Y-Q, Lu M, Tao P-Y, Zhang Y-J, Gong X-T, Zhang G-Q, Li H-L and Yang Z 2016 J. Power Sources 307391
[12] Ong L K, Kurniawan K, Suwandi A C, Lin C X, Zhao X S and Ismadji S 2012 Progress in Nat. Sci. Mat. Int. 22624
[13] Hema M and Arivoli S 2007 Int. J. Phys. Sci. 2010
[14] Taer E and Taslim R 2018 AIP Conf. Proc. 1927030006-1
[15] Taer E, Apriwandi, Yusriwandi, Mustika W S, Zulkifli, Taslim R, Sugianto, Kurniasih B, Agustino and Dewi P 2018 AIP Conf. Proc. 1927030036-1.
[16] Taer E, Sugianto, Sumantre M A, Taslim R, Iwantono, Dahlan D and Deraman M 2014 Adv. Mat. Research 89666
[17] Iwantono, Taer E and Umar A A 2012 AIP Conf. Proc. 1454251
[18] Carrott P J M, Nabais J MV, Carrott, MML R and Pajares, JA 2001 Carbon 391543
[19] Cullity B D 2001 Elements of X-Ray Diffraction, Ed. 3, Amazon Prentice Hall
[20] Li F, Chi W, Shen Z, Wu Y, Liu Y and Liu H 2010 Fuel Process Technol. 9117
[21] Deraman M, Daik R, Soltaninejad S, Nor N S M, Awitdrus, Farma R, Mamat N F, Basri N H and Othman M A R 2015 Adv. Mat. Research 11081
[22] Li L, Liu E, Li J, Yang Y, Shen H, Huang Z, Xiang X and Li W 2010 J. Power Sources 1951516.
[23] Taer E, Dewi P, Sugianto, Syech R, Taslim R, Salomo, Susanti Y, Purnama A, Apriwandi, Agustino, Setiadi R N 2018 AIP Conf. Proc. 1927030026-1
[24] Farma R, Deraman M, Awitdrus A, Talib I A, Taer E, Basri N H, Manjunatha J G, Ishak M M, Dollah B N M and Hashmi S A 2013 Bioresour. Technol. 132254
[25] Taer E, Deraman M, Talib I A, Awitdrus A, Hashmi S A and Umar AA 2011 Int. J. Electrochem. Sci. 63301
[26] Taer E, Taslim R, Mustika W S, Kurniasih B, Agustino, Afrianda A and Apriwandi 2018 Int. J. Electrochem. Sci. 138428
[27] Lee, Yi S and Park S J 2013 J. Solid State Chem. 207158
[28] Ra E J, Raymundo-Piñero E, Lee Y H and Béguin F 2009 Carbon 472984