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The effect of microwave irradiation in activated carbon processing from sago waste to physical and electrochemical properties of electrode supercapacitor cells

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Abstract. This study demonstrates the effect of power in microwave irradiation for providing sago waste based activated carbon. The physical characterization was performed for the carbon electrode and electrochemical properties was elucidate for the supercapacitor cells. The preparation of carbon electrodes begins by pre-carbonized, grinding and followed by sifting. The improvement of the pore properties of the carbon sample was carried out by chemical activation using KOH activating agent at a concentration of 0.2 M followed by microwave irradiation at a power variation of 200, 400, 600, 800 watts for 20 minutes. Carbon pellets were formed at a pressure of 8 tons and carbonized at 600 °C in an N\(_2\) gas environment followed by physical activation using water steam at a temperature of 850 °C. The physical properties testing showed 400 watt is the optimum of irradiation power in production of carbon electrode made from sago waste material. This optimum condition shown by the minimum condition on the density and microcrystalline height and maximum condition on the surface area and carbon content. This excellent physical properties caused the optimum electrochemical properties was found in the supercapacitor cell. The optimum specific capacitance is obtained as high as 95 F g\(^{-1}\). As an addition it has also been analyzed the appearance of the surface morphology of the electrode sample.

1. Introduction

One of the most widely researched studies today is storage energy devices such as supercapacitors. Supercapacitors are in demand because of their ability to store considerable energy [1]. One factor that influences the ability to energy storage in a supercapacitor is the material on the electrode [2]. In theory almost all organic materials with a high percentage of carbon can be activated to improve their physical and capacitive properties. However, in practice, the best material for activated carbon must contain a minimum amount of organic material such as lignin and can be produced at a relatively low cost. According to Pope (1999), organic materials that contain lignin, hemicellulose, and cellulose can be used as raw material for prepared of activated carbon [3]. The process of sago flour produces waste containing crude fiber in sago waste which are contains 21% lignin, 20% cellulose, extractive substances and ashes [4]. One study on the use of sago as a supercapacitor application was carried out...
by Aripin et al. 2010 [5]. In his research method, Arifin performed a chemical activation method using KOH solution and physical activation performed at varied temperatures. In addition, the sample electrode was prepared with polytetrafluoroethylene (PTFE) as an adhesive material. From the research, Aripin obtained a specific capacitance value of 16 F g\(^{-1}\) up to 64 F g\(^{-1}\). This research was conducted to prepared monolithic activated carbon from sago waste by chemical activation method using KOH (Potassium Hydroxide) and assisted by microwave irradiation. It is expected that the microwave irradiation can provide better pores on the electrode so as to increase the capacitive properties. The focus of the research is on the variation of microwave radiation power and its effect on the electrochemical measurement properties of sago waste activated carbon electrode.

2. Experimental Method

2.1. The preparation of carbon electrodes

The preparation of carbon electrodes from sago waste biomass is carried out in several steps such as sample preparation, pre-carbonized, chemical activation and assisted by microwave irradiation, pyrolysis process and finally preparation of supercapacitor cell [6]. The basic material used in this study is sago waste biomass (Metroxylon sago) which taken from Centai Village, Merbau Island, Meranti Regency, Riau, Indonesia. The pre-carbonization process is carried out for 2 hours, with a temperature of 100-250 °C. Chemical activation is performed by mixing sago waste powder with 0.2 M KOH solution. The next process, the KOH activated sample is irradiated using a microwave oven with a radiation time of 20 minutes and the power is varied, namely 200, 400, 600 and 800 Watts. Based on this variation, the samples were labeled of AS/200, AS/400, AS/600 and AS/800. The irradiation process aims to assist the chemical activation. Activated carbon powder are converted into pellets using a hydraulic press [7]. The pyrolysis process is carried out using the previous method [8] of flowing N\(_2\) gas at environment at a temperature of 600 °C and Followed by physical activation by flowing water steam at a temperature of 850 °C for 2 hours. The pyrolysis process is generally seen in Figure 1. Finally, the carbon pellet is prepared as a supercapacitor cell. Supercapacitor cells are composed of carbon electrodes, separators, electrolytes and current collectors [1,9]. The separator used is duck eggshell membrane [10] and 1 M H\(_2\)SO\(_4\) as electrolyte [11].

![Figure 1. Pyrolysis process of sago waste carbon electrode](image)

2.2. Characterization of Electrode Physical Properties

Measurement of physical properties of sago waste activated carbon electrodes included of density, crystalline structure, surface morphology and element contents of carbon electrodes. Density is evaluated by measuring the dimensions and mass of the electrodes. Evaluation of the microcrystalline
structure was carried out using X-ray diffraction. X-ray diffraction was used to determine interlayer spacing, microcrystalline dimension and crystalline phase by using standard formulas on carbon electrodes. The diffraction angle used is in the angle range 10° - 100°. SEM is a method used to review the morphological structure of a material sample while EDX is method to determine the chemical content of the carbon electrodes. SEM and EDX measurements are performed using the Jeol JSM 6510 LA instrument.

2.3. Characterization of Electrochemical Properties
Measurement of electrochemical properties of supercapacitor cells using the Cyclic Voltametry method with two electrode systems. Cyclic Voltametry measurements were carried out using the Physics CV UR Rad-Er 5841 with a potential window from 0 to 500 mV and a scan rate of 1 mV s⁻¹.

3. Result and Discussion
3.1. Analysis of density
The density of carbon electrodes have an important role to produce good capacitance. Density is related to the formation of the pore structure and the internal electrode resistance at a electrochemical measurements. Carbon electrodes before and after physical carbonization have different densities for each variety. Table 1 shows that the density has decreased after carbonization and physical activation processes. Irradiation power affects the density, where an increase in the irradiation power of 200 to 400 watts in the AS/200 sample and AS/400 sample has a significant decrease in density with a percentage reduction of 19%. The decreasing in density drastically indicates that at 400 watts there is a decrease in water content, a shrinking of volatile contains and a maximum pore formation. The irradiation power of 600 and 800 watts given to the AS/600 and AS/800 samples also show the decreasing in density but with a lower percentage. This is indicated the higher irradiation power levels causes damage to organic molecules [12]. The lowest density electrode is an indicator to choose the best irradiation power in preparation of carbon electrodes from sago waste and will be followed by other analyzed such as XRD, SEM, and CV in the next subsection.

| Sample codes | Before pyrolysis (gcm⁻³) | After pyrolysis (gcm⁻³) |
|--------------|---------------------------|-------------------------|
| AS/200       | 0.965                     | 0.911                   |
| AS/400       | 0.959                     | 0.776                   |
| AS/600       | 0.886                     | 0.879                   |
| AS/800       | 0.866                     | 0.850                   |

3.2 Analysis of X-ray diffraction
This X-ray diffraction analysis aims to determine the degree of crystallinity of carbon electrode samples. X-ray diffraction curves (XRD) for sago waste carbon electrodes in AS/200 and AS/400 samples are shown in Figure 2. Figure 2 shows the results of X-ray diffraction characterization which shows the presence of two broadening peaks with 2θ angle in the range of 10’ to 100’. These two broadening peaks indicate that the carbon electrode is amorphous structure for carbon material from biomass [13]. These diffraction angles represent the peak position corresponding to the plane (002) and (100) in the carbon structure [6]. The results of X-ray diffraction curves founded the content elements other than carbon as evidenced by the presence of sharp and narrow peaks in X-ray diffraction curve patterns. The microcrystalline width (L₀) and microcrystalline height (Lₜ) parameters determined by using Microcal Origin software to facilitate data processing. The L₀ and Lₜ obtained are calculated based on the standard formulas [14,15,16,17]. The L₀ and Lₜ for both samples can be observed in Table 2. The Lₜ for AS/400 sample is smaller than the Lₜ in the AS/200 sample along with the increased irradiation power given. This is indicated that the irradiation process effect to rearranging carbon atoms. Carbon atom grew larger for horizontal direction and grew smaller for
vertically, so the $L_c$ will be smaller. The $L_c$ can be used as a reference to determine the specific surface area of the sample. Based on the relationship of microcrystalline dimensions ($L_c$) of carbon electrodes to the specific surface area given by the empirical formula $SSA_{xrd} = \frac{2}{(ρ_{xrd} L_c)}$ [18, 19]. The specific surface area for AS/400 samples as high as 936.114 m$^2$g$^{-1}$ higher than AS/200 samples of 844.594 m$^2$g$^{-1}$. This specific surface area are similar as the electrode surface area of other materials, such as coffee shell [20] and cassava peel waste [21].

**Table 2.** Diffraction angle ($2θ$), microcrystallinity height ($L_c$) and microcrystallinity width ($L_a$) of sago waste carbon electrode

| Sample codes | $2θ_{(002)}$ (°) | $2θ_{(100)}$ (°) | $L_c$ (Å) | $L_a$ (Å) |
|--------------|------------------|------------------|-----------|-----------|
| AS/200       | 24.518           | 44.118           | 11.364    | 21.776    |
| AS/400       | 24.961           | 44.568           | 10.116    | 22.200    |

**3.3 Analysis of scanning electron microscopy**

The surface morphology of the samples was analyzed by using the SEM method which is presented in Figure 3. Figure 3(a) and 3(b) are surface morphology for AC/200 samples while Figure 3(c) and 3(d) are for AC/400 sample. Figure 3(a) shows a smaller and smoother particle structure and a brighter color. The color brightness indicates that the sample has no clear pores. The size of the particle is in range of 1.95-4.23 µm. Figure 3(b) shows a sample with 40000 magnification more clearly visible, the small holes formed between particles and the size of the carbon particles formed have a range of 0.22-0.39 µm. Figure 3(c) shows more dense and smaller surface particles on carbon electrodes. The size of the particles formed is in ranges of 1.93-3.40 µm. Figure 3(d) shown the sample with 40000 magnification more clearly the pores between particles and the formation of pore cracks and has a darker color. The size of carbon particles formed has a large range from 0.28 to 0.68 µm. Based on variations in the power of microwave irradiation used showed differences in sample morphology. The particle size in the AS/400 sample is smaller than the AS/200 sample. The smaller particle size is caused by the higher irradiation power made the pores to become wider due to the loss of part of the cavity which is enclosed in carbon particles so that it forms good pores.
3.4 Analysis of Energy Dispersive X-Ray

Table 3. The chemical content for AS/200 and AS/400 samples

| Chemical content | AS/200 Mass % | AS/200 Atom % | AS/400 Mass % | AS/400 Atom % |
|------------------|--------------|--------------|--------------|--------------|
| C                | 90.23        | 93.54        | 92.77        | 95.78        |
| O                | 7.01         | 5.45         | 4.07         | 3.16         |
| Mg               | 0.20         | 0.10         | 0.23         | 0.12         |
| Si               | 0.83         | 0.38         | 0.28         | 0.12         |
| K                | 0.28         | 0.09         | 0.68         | 0.22         |
| Ca               | 1.41         | 0.44         | 1.97         | 0.61         |
| Totals           | 100%         |              |              |              |

The Energy X-Ray Dispersive is used to determine element contents in AC/200 and AC/400 activated carbon samples. The element contents in sago waste carbon electrode are carbon (C), oxygen (O), magnesium (Mg), silicon (Si), calcium (Ca), and potassium (K) as shown in Table 3. Based on Table 3, the element contents in the carbon electrode samples are dominated by carbon (C) with a percentage range of 90% to 95%. The carbon content obtained in this study is also similar to the previously reported carbon content with different carbon materials, such as from durian shell [22] and banana stems [23]. Based on this data shows that the microwave irradiation process has an important role in increasing the amount of carbon content. In addition to carbon, the oxygen element has the second highest percentage in the sample. This is indicated when pyrolysis process, the oxygen content in carbon samples is not completely decomposed or bonding occurs in the activation process. Meanwhile, the presence of the element potassium (K) because the KOH as activator agents are not neutralized perfectly. While the presence of elements of calcium (Ca) and silica (Si) is caused by the chemical content in sago waste. Based on variations in microwave irradiation power carried out on the sample, it gives a difference in the carbon elements produced. This is possible because when too much
irradiation is applied to the sample, the higher the temperature is produced and the more non-carbon material evaporates. According to Marsh and Reinoso (2006) the carbon content amounted to 90% when heated at a temperature of 927 °C and 99% carbon content when heated at a temperature of 1327 °C and depending on the biomass materials [24].

3.5 Analysis of capacitive properties
The Cyclic Voltammetry (CV) was carried out to determine the specific capacitance of the supercapacitor cell. The CV measurements for supercapacitor cell were carried out at a potential window of 0 to 0.5 Volts with a scan rate of 1 mV s\(^{-1}\) [25]. The CV result shown a curve between current density (Acm\(^{-2}\)) and voltage (V) as shown in Figure 4. The charge and discharge current affects on the shape of the resulting curve. The wider curve indicated the greater of charge and discharge current which results the higher of specific capacitance, while the lower of charge and discharge current indicates the smaller specific capacitance. Figure 4 shows that the AS/400 sample presents a much larger curve area, showing greater performance of electrochemical cell [26]. The specific capacitance (Csp) can be calculated by using standard equations. The Csp was obtained for AS/200, AS/400, AC/600 and AC/800 are 61.019 Fg\(^{-1}\), 94.975 Fg\(^{-1}\), 36.567 Fg\(^{-1}\) and 9.435 Fg\(^{-1}\), respectively. The specific capacitance in this study has a greater than arpin reported that using the same biomass material with specific capacitance of 64 Fg\(^{-1}\) [5]. The decrease in specific capacitance in AS/600 and AS/800 samples because of the higher irradiation power will cause the higher formation of ash, so that the pores on the carbon structure experience widening, damage and finally reduce the specific capacitance. The specific capacitance against irradiation power is shown in Figure 5.

![Cyclic voltamogram for all samples](image1)

![Relationship of power and specific capacitance](image2)

4 Conclusion
The effect of power in microwave irradiation for providing sago waste based activated carbon has been studied. Irradiation power affects to get the best physical and electrochemical properties. The 400 watts is the optimum of irradiation power in production of carbon electrode made from sago waste material. This optimum condition shown by the lowest of density, highest specific surface area and maximum condition on the carbon content. The 400 watts irradiation power produce the carbon electrode sample with lowest density of 0.850 gcm\(^{-3}\), high surface area of 936.114 cm\(^2\)g\(^{-1}\), the highest carbon content is 95% and optimum specific capacitance as high as 95 Fg\(^{-1}\).

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References

[1] González A, Goikolea E, Barrena J A, Mysyk R 2016 Renewable and Sustainable Energy Reviews 58 1189
[2] Abioye A M, Ani F N 2015 Renewable and Sustainable Energy Reviews 52 1282
[3] Pope J P 1999 Civil Engineering Dept. Virginia Tech. USA.
[4] Vikineswary S, Shim Y L, Thambirajah J J, Blake-Brough N 1994 Resour. Conserv. Recycl. 11289
[5] Arpin H, Lestari L, Ismail D, Sabchevski S, The Open Materials Science Journal, 4117
[6] Taer E and Taslim R 2018 AIP Conf. Proc. 1927 020004-1
[7] Taer E, Deraman M, Talib A, Awitdrus A, Hashmi SA and Umar AA 2011 Int. J. Electrochem. Sci. 63301
[8] Taer E, Apriwandi, Yusriwandi, Mustika W S, Zulkiﬁli, Taslim R, Sugianto, Kurniasih B, Agustino and Dewi P 2018 AIP Conf. Proc. 1927030036-1
[9] Itagaki M, Suzuki S, Shitanda I, Watanabe K, Nakazawa H 2007 J. Power Sources 164 415
[10] Taer E, Sugianto, Sumantre M A, Taslim R, Iwantono, Dahlan D and Deraman M 2014 Adv. Mat. Research 89666
[11] Iwantono, Taer E, Umar A A 2012 AIP Conf. Proc. 1454 251
[12] Saifuddin N, Chua KH 2004 Malaysian Journal of Chemistry 077
[13] Yu G, Lei L, Yuming J, Yu W, Chuanjun Y, Yingjin W, Gang C, Junjie G, Haiyan L 2015 Appl. Energy 153 41
[14] Carrott P J M, Nabais J M V, Carrott M M L R, Pajares J A 2001 Carbon 39 1543
[15] Cullity B D 2001 Elements of X-Ray Diffraction Ed. 3 amazon Prentice Hall.
[16] Awitdrus, Deraman M, Talib I A, Omar R, Jumali M H, Taer E, Saman M H 2010 Sains Malaysiana 39 83
[17] Nabais J M V, Teixeira J G, Almeida I 2010 Bioresource Technol. 102 2781
[18] Shoai b M, Al-Swa idan H M 2015 Biomass and bioenergy. 73 124
[19] Deraman M, Daik R, Soltaninejad S, Nor N S M, Awitdrus, Farma R, Mamat N F, Basri N H, Othman M A R 2015 Adv. Mater. Research 1108 1
[20] Jisha M R, Hwang Y J, Shin J S, Nahm K S, Kumar T P, Karthikeyan K, Dhanikaivelu N, Kalpana D, Renganathan N G, Stephan A M 2009 Mater. Chem. and Phys. 11533
[21] Ismanto A E, Wang S, Soetaredjo F E, I smadij S 2010 Bioresource Technol. 101 3534
[22] Taer E, Dewi P, Sugianto, Syech R, Taslim R, Salomo, Susanti Y, Purnama A, Apriwandi, Agustino, Setiadi R N 2018 AIP Conf. Proc. 1927 030026-1
[23] Taer E, Taslim R, Mustika W S, Kurniasih B, Agustino, Afrianda A and Apriwandi 2018 Int. J. Electrochem. Sci. 138428
[24] Marsh H, Rodrigues-Reinoso F 2006. Activated Carbon. Elsevier Science and Technology Book Netherlands
[25] Inagaki M, Konno H, Tanaike O 2010 J. Power Sources 195 7880
[26] Song K, Ni H, Fan L-J 2017 Electrochem. Acta 235 233