Preliminary study of activated carbon from water chestnut (Eleocharis dulcis)

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Abstract. Preliminary study of activated carbon from water chestnut (Eleocharis dulcis) has been done. The water chestnut plants, which were taken from Barito Kuala regency, South Kalimantan, were carbonized at 400 °C. The carbonized time were varied in 1 and 2 hours. Then the carbons were activated by KOH and H2SO4 solutions. The activated time were also varied in 1 and 2 hours. The physical-chemical properties of water chestnut charcoal, such as water content, ash content, volatile and fixed carbon values, before and after activated, were characterized. The results showed that the water content, ash content, volatile and fixed carbon values of activated carbon were in the range of 5.63-12.17%; 10.95-17.22%, 41.78-56.18% and 34.66-45.05%, respectively.

Keywords: Water content, ash content, volatile, fixed carbon.

1. Introduction
Activated carbon is an amorphous compound produced from carbon-containing materials and has high adsorption ability. The adsorption properties of activated carbon are depended on its surface porosity. While the surface area, dimensions and distribution of carbon atoms composing the active charcoal structure are highly dependent on the raw material, carbonization conditions and activation process [1]. In making activated carbon, raw materials should have contained carbon [2]. One of them is biomass. Many materials, such as rice husk [3], pineapple leaf [4], have been used to make activated carbon. Several researches in producing activated carbon from aquatic plants were already done. The plants used were water chestnut [5], cattail [6], hyacinth water [7, 8] and seedpod lotus [9]. These activated carbons have been developed for supercapacitor electrode applications.

Water chestnut (Eleocharis dulcis) or purun tikus (in local language) grows in swamp areas in South Kalimantan. The growth of water chestnut (WC) is quite fast. This caused WC was categorized as weed [10]. As aquatic plant, WC has quite high carbon content, about 50.68%, as investigated by Maftu’ah [11]. The use of WC plant in South Kalimantan is still limited such as food for swamp buffalo, making mats [10] and biofilter [12]. In other researches, WC has been also used as composite materials [13].

The quality of activated carbon depends on several factors such as carbonization temperature, carbonization time, activator, activation time and particles size [3, 4, 7, 14, 15]. Research conducted by Rohmah [3] showed that longer carbonization time gave higher adsorption coefficient. Activation process was also an important factor to activated carbon. Activation can be done both chemically and physically. Physics activation is done to expand the pore of activated carbon with the help of heat, steam and CO2 gas. While chemical activation used chemicals as activators, such as alkali metal...
hydroxides, chlorides, sulfates, phosphates from alkaline earth metals and inorganic acids [16]. Setiawan [4] showed that activator H$_2$SO$_4$ gave highest adsorption coefficient.

In this study, the effect of carbonization time and different activator (KOH and H$_2$SO$_4$) to WC charcoal would be investigated. The activated carbons were used as heavy metal adsorbent. Therefore, in this article, the preliminary study of activated carbon from WC would be investigated especially its’ physical-chemical properties such as water content, ash content, volatile and fixed carbon. The measurement would follow SNI No. 06-3730-1995 [17] about quality and testing of activated carbon. SEM-EDX would be used to investigate the porous, morphology and elements of activated carbon.

2. Experimental Methods

In this research, water chestnut plants were taken from Barito Kuala regency, South Kalimantan. The WCs were dried and cut into pieces, about 2 cm long and then carbonized at 400 °C. The carbonized times were varied in 1 and 2 hours. Then the WC charcoals were crushed and filtered to sizes of 60 and 120 mesh. Then samples were activated by KOH and H$_2$SO$_4$. Some physical characteristics, such as water content, ashes content, volatile and fixed carbon, would be examined according to SNI No. 06-3730-1995 [9].

To measure the water content, the sample was put into oven at temperature 105°C for 3 hours. Then it was put in desiccators till cooled down. Initial and relative changes in weight of the samples were measured. Percentage water content was calculated using the following equation:

\[
\text{Water Content (\%)} = \frac{M_1-M_2}{M_1} \times 100\% 
\]  

(1)

where $M_1$ is the Initial weight of sample and $M_2$ is the Final weight of sample after drying.

Percentage of ash content of sample was investigated also according to SNI No. 06-3730-1995 [9]. The sample was put into the oven at temperature 850°C for 4 hours till all samples turn into ashes. The samples were cooled down in desiccator before measuring the relative change in weight. Percentage of ash content was calculated using the following equation:

\[
\text{Ash Content (\%)} = \frac{M_1-M_2}{M_1} \times 100\% 
\]  

(2)

where $M_1$ is the Initial weight of sample and $M_2$ is the Final weight of sample.

The sample volatile was investigated by putting the sample into the oven at temperature 950°C for 7 minutes then the relative change in weight was measured. Percentage of volatile content was calculated using the following equation:

\[
\text{Volatile (\%)} = \frac{M_1-M_2}{M_1} \times 100\% 
\]  

(3)

where $M_1$ is the Initial weight of sample and $M_2$ is the Final weight of sample after heating.

The fixed carbon values could be calculated from ash content and volatile using equation:

\[
\text{Fixed Carbon (\%)} = 100\% - (\text{Volatile (\%)} + \text{Ash Content (\%)}) 
\]  

(4)

While the morphologies and elements would be analyzed from SEM-EDX.
3. Results and discussion

3.1. Physical properties of water chestnut before activation

The obtained value of water content, ash content, volatile and fixed carbon of WC before and after carbonization are presented in Table 1.

| Particles Size | Carbonisation time | Water Content (%) | Ash Content (%) | Volatile (%) | Fixed Carbon (%) |
|----------------|--------------------|-------------------|----------------|--------------|-----------------|
| 60 Meshs       | Not Carbonized Yet | 9.83              | 15.33          | 45.17        | 39.50           |
|                |                    | 9.53              | 14.79          | 45.24        | 39.97           |
|                | Average            | 9.72              | 15.07          | 45.38        | 39.55           |
| 120 Meshs      |                    | 9.53              | 15.10          | 45.73        | 39.17           |
|                | Average            | 8.96              | 13.97          | 40.65        | 45.38           |
| 60 Meshs       | 1 hour             | 3.64              | 13.15          | 44.33        | 42.52           |
|                | Average            | 4.06              | 13.67          | 44.40        | 41.93           |
| 120 Meshs      |                    | 3.58              | 12.85          | 38.49        | 48.99           |
|                | Average            | 4.00              | 12.79          | 38.46        | 48.75           |
|                | 2 hours            | 3.56              | 12.19          | 36.99        | 50.82           |
|                | Average            | 3.59              | 12.06          | 37.35        | 50.59           |
|                | 120 Meshs          | 3.95              | 12.39          | 37.15        | 50.47           |
|                | Average            | 3.70              | 12.21          | 37.16        | 50.63           |
|                |                    | 3.75              | 11.48          | 35.26        | 53.25           |
|                | 120 Meshs          | 3.53              | 11.78          | 35.39        | 52.83           |
|                | Average            | 3.58              | 11.59          | 35.19        | 53.22           |

As can be seen in Table 1, the average of water content values of WC before and after carbonization were consistently decreased as the particles size become smaller. When the particles size become smaller, the water could be easier evaporate. Therefore the water content would be smaller. The ash content and volatile were also decreased. When the particles are smaller, almost all the samples would be burned perfectly and left a small amount of ashes. The heat would also burn the others compound such as, cellulose and lignin, and leave the carbon (fixed carbon).

In Table 1, it can also be seen that the water content, ash content and volatile of WC before carbonization were larger as compare to the same properties of WC after carbonization. It seems that the longer of carbonization time would give smaller values of water content, ash content and volatile. The exception for fixed carbon, the longer carbonization time seems increase the carbon content in the samples.
3.2. Physical properties of water chestnut after activation

The obtained value of water content, ash content, volatile and fixed carbon of WC after carbonization and activation are presented in Table 2. As can be seen in Table 2, samples were carbonized in two different times, 1 hour and 2 hours. Then both of samples were activated by two different activators, which are KOH and H$_2$SO$_4$. The average of water content values of WC were, as in Table 1 above, consistently decreased as the particles size become smaller. Consistency could be seen also for carbonization time where longer of carbonization time would give smaller values of water content, ash content and volatile.

### Table 2. Physical properties of water chestnut after carbonization and activation.

| Carbonisation Time | Activator | Particles Size | Water Content (%) | Ash Content (%) | Volatile (%) | Fixed Carbon (%) |
|--------------------|-----------|----------------|-------------------|----------------|--------------|-----------------|
| 1 hour             | KOH       | 60 Meshs       | 17.15             | 10.98          | 54.46        | 34.56           |
|                    |           |                | 17.12             | 10.89          | 54.42        | 34.68           |
|                    |           |                | 17.08             | 10.99          | 54.27        | 34.74           |
|                    |           | Average        | 17.12             | 10.95          | 54.39        | 34.66           |
|                    |           | 120 Meshs      | 12.14             | 13.60          | 48.74        | 37.66           |
|                    |           |                | 12.16             | 13.54          | 48.78        | 37.69           |
|                    |           | Average        | 12.17             | 13.56          | 48.78        | 37.66           |
|                    |           | 60 Meshs       | 5.51              | 12.49          | 42.49        | 45.02           |
|                    |           |                | 5.89              | 12.46          | 42.46        | 45.08           |
|                    |           | Average        | 5.63              | 12.47          | 42.48        | 45.05           |
|                    | H2SO4     | 120 Meshs      | 8.29              | 16.09          | 41.85        | 42.06           |
|                    |           |                | 8.15              | 16.15          | 41.78        | 42.07           |
|                    |           | Average        | 8.23              | 16.17          | 41.78        | 42.05           |
|                    |           | 60 Meshs       | 12.26             | 12.74          | 52.21        | 35.05           |
|                    |           |                | 12.22             | 12.49          | 52.23        | 35.27           |
|                    |           | Average        | 12.23             | 12.63          | 52.27        | 35.10           |
|                    |           | 120 Meshs      | 11.07             | 15.56          | 46.42        | 38.01           |
|                    |           |                | 11.13             | 15.63          | 46.39        | 37.99           |
|                    |           | Average        | 11.07             | 15.59          | 46.37        | 38.04           |
|                    |           | 60 Meshs       | 10.38             | 13.11          | 42.31        | 44.58           |
|                    |           |                | 10.36             | 13.21          | 42.29        | 44.50           |
|                    |           | Average        | 10.38             | 13.14          | 42.28        | 44.58           |
|                    |           | 120 Meshs      | 7.78              | 17.19          | 42.27        | 40.54           |
|                    |           |                | 7.73              | 17.08          | 42.11        | 40.80           |
|                    |           | Average        | 7.76              | 17.22          | 42.16        | 40.62           |

SNI No. 06-3730-1995 < 10% < 10% < 25% > 65%

In table 2, the average of water content values of WC after activation by KOH and/or H$_2$SO$_4$ were consistently decreased as the particles size become smaller. Activator H$_2$SO$_4$ gave smaller water content values as compare to KOH. Chemically, activator is a process of breaking the carbon chains of...
organic compounds. The activator KOH and H$_2$SO$_4$ would clean the charcoals which were immersed to them, from the residue of the carbonization process. Almost all samples of WC which were activated by H$_2$SO$_4$ were fulfilled SNI No. 06-3730-1995 (<10%). While all samples of WC which were activated by KOH were not fulfilled SNI No. 06-3730-1995 (<10%). This is in line with Setiawan [4] which claimed that H$_2$SO$_4$ gave a better result for activated carbon from pineapple leaf.

3.3. EDX analysis of water chestnut
EDX analysis of water chestnut at sizes of 60 and 120 mesh, before and after carbonization are presented in Figure 1. Figure 1(a) and (b) show SEM and EDX measurements of WC at size of 60 and 120 mesh before carbonization. In Figure 1 (a), the carbon element contained in WC powder at 60 mesh particle size was 36.71% of weight while at size 120 mesh (Figure 1(b)) was 42.34%. The carbon element has the highest concentration in both sizes. These results could be referred to Maftu’ah’s research [11] which wrote that WC has quite high carbon content, about 50.68%. Figure 1 (c) and (d) show SEM and EDX measurements of WC at size of 120 mesh which were carbonized for 1 and 2 hours. The carbon element in Figure (c) was 49.25% which is larger than carbon in Figure 1 (b). This result was in line with research conducted by Rohmah [3]. Carbonization process burned some organic materials such as cellulose and lignin, and increases the carbon concentration. Comparing Figure 1 (b), (c) and (d), it can be seen that the cellulose textures were collapse after carbonization. From Figure 1 (d), it could be concluded that 2 hours carbonization gave more carbon concentration.
Figure 1. EDX analysis of water chestnut at sizes of: (a) 60 mesh, not carbonized, (b) 120 mesh, not carbonized, (c) 120 mesh, 1 h carbonization and (d) 120 mesh, 2 h carbonization.

4. Conclusions
As conclusions, water content, ash content, volatile and fixed carbon values of water chestnut before carbonization were in the range of 8.96 - 9.72%; 13.97-16.07%; 40.65-45.38% and 39.55-45.38%, respectively. While the water content, ash content, volatile and fixed carbon values of the activated carbon were in the range of 5.63-12.17%; 10.95-17.22%, 41.78-56.18% and 34.66-45.05%, respectively. Smaller particles size and longer carbonization time would give smaller value of water content, ash content and volatile, except for fixed carbon. The water content of WC which were activated by H$_2$SO$_4$ were fulfilled SNI No. 06-3730-1995 (<10%). While all samples of WC which were activated by KOH were not fulfilled SNI No. 06-3730-1995 (<10%).

5. References
[1] Kyotani T 2000 Control of Pore Structure In Carbon Carbon 38:269-286.
[2] Martin A 2010 Adsorpsi isothermal karbon dioksida dan metana pada karbon aktif berbahan dasar batubara sub bituminus indonesia untuk pemurnian dan penyimpanan gas alam Disertasi Departemen Teknik Mesin Fakultas Teknik, Universitas Indonesia.
[3] Rohmah PM & Redjeki AS 2014 Pengaruh waktu karbonisasi pada pembuatan karbon aktif berbahan baku sekam padi dengan aktivator KOH Jurnal Konversi 3(1):19-26.
[4] Setiawan AA, Shofiyani A, & Syahbanu I 2017 Pemanfaatan limbah daun nanas (Ananas comosus) sebagai bahan dasar arang aktif untuk adsorpsi Fe (II) Jurnal Kimia Khatulistiwa 6 : 66-74.
[5] Zulkifli, Awitdrus and Taer E 2018 Studi awal pemanfaatan purun tikus sebagai elektroda superkapasitor menggunakan aktivasi uap air J. Aceh Phys. Soc. 7(1): 30-34.
[6] Yu M, Y Han, J Li, L Wang 2016 CO$_2$-activated porous carbon derived from cattail biomass for removal of malachite green dye and application as supercapacitors Chemical Engineering Journal 17:1-41.
[7] Sangkota VDA, Supriadi, & Said I 2017 Pengaruh aktivasi kimia arang tanaman eceng gondok (Eichhornia crassipes) terhadap adsorpsi logam timbal (Pb). Jurnal Akademika Kimia 6 (1) : 48-54.
[8] Kurniawan F, Wongso M, Ayucitra A, Soetaredjo FE, Angkawijaya AE, Ju YH, Ismadji S 2014 Carbon microsphere from water hyacinth for supercapacitor electrode Journal of the Taiwan Institute of Chemical Engineers 47: 197-201.
[9] Liu B, Zhou X, Chen H, Liu Y, Li H 2016 Promising porous carbons derived from lotus seedpods with outstanding supercapacitance performance Electrochimica Acta 208 :55–63.
[10] Asakin S & M Thamrin 2012 Manfaat purun tikus (Eleocharis dulcis) pada ekosistem sawah rawa Jurnal Litbang Pertanian 31(1): 35-42.
[11] Prihatini NS, Krisdianto, Setyorini A, Azizah N, Khameni S, Astuti DT 2011 Potensi purun tikus (Eleocharis dulcis) sebagai biofilter Proceedings Environmental Talk: Toward A Better
Green Living.

[12] Wardhana H & Ninis H Haryanti 2017 The characteristics of purun tikus particle board cement board *IOSR Journal of Applied Chemistry (IOSR-JAC)* 10 (1):01-4.

[13] Wibowo S, Syafii W & Pari G 2010 Karakteristik Arang Aktif Tempurung Biji nyamplung (Calophyllum inophyllum Linn) *Jurnal Penelitian Hasil Hutan* 28(1):43-45 DOI 10.20886/jphh.2010.28.1.43-54.

[14] Reyra AS, Daud S & Yenti SR 2017 Pengaruh Massa dan Ukuran Partikel Adsorben Daun Nanas Terhadap Efisiensi Penyisihan Fe Pada Air Gambut *Jurnal Online Mahasiswa (JOM) FTeknik* Vol. 4 Universitas Riau.

[15] Triyanto A 2013 Peningkatan kualitas minyak goreng bekas menggunakan arang ampas tebu teraktivasi dan penetralan dengan NaHSO₃ Tugas Akhir Jurusan Kimia Fakultas MIPA Universitas Negeri Semarang.

[16] SNI 06-3730-1995: Arang Aktif Teknis Dewan Standarisasi Nasional Jakarta.

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