Preparation of Activated Carbon from Durian Shell and Seed

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Abstract. This study presents the preparation of activated carbon from durian shell (DShAC) and durian seed (DSeAC) based by chemical activation with potassium hydroxide (KOH) as an activating agent under the nitrogen flow. In order to find the optimum physical characteristics, variation in concentration of KOH, carbonization temperature and duration time was employed. The result shows that activated carbon from durian waste is a promising activated carbon as the highest yield was obtained from the carbonization process that occurs at 400°C for four hours with the KOH concentration is 0.6 M.

1. Introduction

Activated carbon, also known as activated charcoal is a form of carbon that has been processed to produce porous structure and large surface area which is suitable for adsorption or chemical reactions. Today, activated carbon is identified as an effective adsorbent produced from a variety of carbonaceous source material. This is due to the common unique characteristic of activated carbon which has complex pore structure, large specific surface area, good chemical stability, and various oxygen-containing functional groups on the surface [1]. These excellent features have made it broadly used in environmental protection, water and wastewater treatment, gas filters, and the purification recovery of chemicals in industry [2, 3]. The pore structure of activated carbon plays an important role because the difference in pore size and shape will affect the capability of adsorption. This is the main criteria by which carbons are selected for a specific application. The adsorption takes place in micropores and mesopores with macropores acting as transport channel. In recent years, there is a growing interest in the application of activated carbons with high proportion of mesoporous structures, which are very suitable for large molecule adsorption, including battery capacitors, catalyst supports, biomedical engineering, and adsorbents for bulky pollutants. However, due to their high production cost, activated carbons tend to be more expensive than other adsorbents and influence engineers' decision for not choosing activated carbon for their plant even though it will give a better performance [4]. To overcome the costing issue, researchers nowadays start to study on the development of low-cost adsorbent which is by using the waste as the raw material as the source of activated carbon such as coal, coconut shell, sawdust agricultural wastes and plant materials and including agricultural industrial by-products. Nevertheless, there is still less of research on production of activated carbon extracted from durian.

Generally, durian is a tropical fruit where the species is from Malvales order in Bombaceae family and genus of Durio with the scientific name is *Durio zibethinus Murray*. This is a most popular seasonal fruit in South East Asia countries and widely known as "King of Fruit" in Malaysia [5]. According to Ahmad Ishak, the Head of Malaysia's Federal Agriculture Marketing Authority, said that the country produces about 300,000 metric tons of durians a year, mainly for domestic consumption. By referring to Department of Agriculture Malaysia, (2009) which is approximately 376,273 metric tonne of durian are produced in year 2008 in Malaysia. This makes Malaysia as also the largest exporter of fresh durian with 34, 904 metric tonne exported in 2001. Since durian consumption result in loads of waste, it would be an advantage if the waste could be processed into valuable product. In other words, there is lots of biomass component generated from durian peels and durian shell that can be re-used to produce the low cost of activated carbon. By

manipulating the waste from durian, more economic value can be derived. Thus, the potential problems that may come from the waste disposal such as respiratory disease which may apart from their pungent smell can be avoided [6]. Wide varieties of activating agent are known such as $ZnCl_2$, H_3PO_4 , and phosphoric acid while the use of potassium hydroxide (KOH) has became the popular studies nowadays [7]. Therefore study investigates on physical characteristic, synthesise and optimum conditions in producing DShAC and DSeAC by using KOH as an activating agent.

2. Methodology

2.1. Material

In this experiment, the durian shell and seed were acquired from the commercial market in Perak. The durian shell and seed was cleaned by washing them with water and dried under the sun for 24 hours in order to remove any dust or inorganic impurities. Drying process was continued in the oven at 80°C for 24 hours. The durian shell and seed were grinded and sieved to desired geometric particle size which is 4 mm in size using grinder.

2.2. Preparation of Activated Carbon

The 4 mm dried durian shell and seed are well mixed with water and KOH solution with the KOH concentration of 0.3 M, 0.45 M and 0.6 M. Then the water was evaporated at 130° C for 24 hours in a sealed ceramic oven. The next process is the carbonization steps. It was heated from room temperature to 400°C, 500°C and 600°C in tube furnace. The flow of N₂ at 600 ml/min volumetric flow rate is maintained throughout the heating process at different duration of 1, 2.5, and 4 hours. This heat treatment steps promote decompositions of durian shell and seed to porous carbonaceous materials and hydrocarbon compound.

2.3. Characterization of Activated Carbon

The grinded shell and seed were characterised by X-Ray Diffraction Analysis (XRD) (Model: Bruker AXS D8-Advance diffractometer) with Cu K α radiation of wavelength, $\lambda = 0.15406$ nm, and operating voltage and current at 40kV and 40 mA, respectively. The pore structure characteristic and the particles shape of activated carbon for the raw material before activation and also after the activation were observed by Field Emission Scanning Electron Microscopy (FESEM, Model Veiss SUPRA 55VP) at accelerating voltage of 0.02 to 30 kV.

3. Results and Discussion

3.1. Yield of Activated Carbon

The percentage of yield weight properties that obtained from the production of activated carbon from durian shell (DShAC) and durian seed (DSeAC) at variation of temperature, process duration and concentration of activating agent was shown in Table 1. Relatively, the high product yields are required in order to produce the commercial activated carbon. In this study, 21.50% to 52.74% of yield is produced by the DShAC while the yields of DSeAC are higher than DShAC which is in range from 25.43% to 55.37%. The yield may influence by the original nature of the raw material. Durian seed may contain higher carbon percentage compare to durian shell. Fig. 1 presents the effect of activation temperature on the yield of activated carbon prior to durian shell (DShAC) and durian seed (DSeAC) at 2.5 hours. As seen in Fig. 1 the yield of both DShAC and DseAC were found to decrease with increasing the carbonization temperature at 2.5 hours. This is due to the higher weight loss rate since the initial large amount of volatiles that can be easily released at higher temperature as well as the loss of moisture to lesser extent [7]. The increasing of temperature helps to release more organic component, leaving the surface less occupied by deposit and reduces the production of yield.

Temp	Time	М	Weight (%)	
(°C)	(hr)		DShAC	DSeAC
400	1.0	0.30	50.66	48.01
400	2.5	0.45	44.92	50.13
400	4.0	0.60	52.74	55.37
500	4.0	0.30	31.95	34.24
500	1.0	0.45	21.50	36.43
500	2.5	0.60	34.30	37.03
600	2.5	0.30	21.54	25.43
600	4.0	0.45	29.20	31.25
600	1.0	0.60	32.40	35.30

Table 1. Weight yield for DShAC and DSeAC

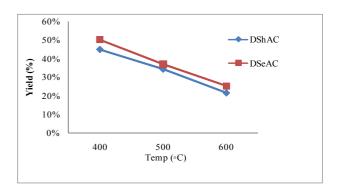


Fig.1: Percentage of yield affected by temperature at 2.5 hours.

Another consideration is a concentration of the activating agent that gives significant effect on the production of the yield. Higher concentration of activating agent will increase the production of the yield. In the presence of chemical agent during the activation promotes depolymerization, dehydration, and redistribution of constituent biopolymer and also favoring the conversion of aliphatic to aromatic compound and influence the yield production [8].

3.2. Surface area and pore structure

3.2.1. Durian shell activated carbon (DShAC)

Before proceed to chemical activation of durian shell, the raw durian shell that has been grinded was sent to the FESEM lab to study the structure of the sample before it was activated. Fig. 2 illustrates the result of Field Emission Scanning Electron Microscopic (FESEM) on the dried durian shell. Before undergoing any activation process, there are not many pores on the surface of the raw durian shell. It can be observed that for raw material, the surface exhibited many thin sheets or layers with large pores within the structure. However, different morphology was obtained after the carbonization process as shown in Fig. 3 and Fig. 4.

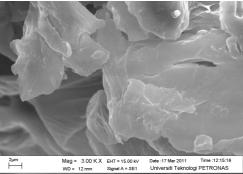


Fig. 2: FESEM for raw durian shell

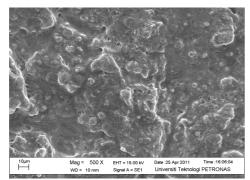


Fig. 3: FESEM for DShAC - 0.45 M of KOH, 600°C for 4 hours

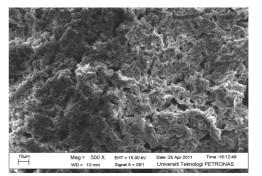


Fig. 4: FESEM for DshAC - 0.60 M of KOH, 400^oC, for 4 hours

Electron micrograph scanning of the activated carbons obtained from the durian shell after 0.6 M of acid treatment followed by 400°C for 4 hours (Fig. 4) heat treatment showed a well-developed porous structure compare to the raw durian shell without any treatment. There are mixtures of meso and micropores in the structure. By comparing the FESEM results between Fig. 3 and Fig. 4, there are many differences can be observed on the surface of both SEM results. The structure is not well developed for the sample that treated using 0.45 M of KOH at 600°C (Fig. 3). This is because the structure collapsed when it reaches high temperature activation. Based on the raw dried durian FESEM results, the surface are smoother as compare to the FESEM results from DShAC. Meanwhile, the results from DShAC are more likely to form a porous surface compare to the dried durian FESEM results. This prove that durian shell can be a promising activated carbon because the more porous the surface, the better DShAC will be produced.

3.2.2. Durian seed activated carbon (DSeAC)

Based on Fig. 5 FESEM result of raw dried durian seed, it can be observed that the seed has a unique hexagon structure on the surface of it. Thus, it is very interesting to study the structure of the surface after activation. By comparing the result on FESEM for the raw durian seed between the carbonize durian seed at 600°C, for 1 hours and at 0.6 M of KOH (Fig. 6) and at 600°C, for 2.5 hours and at 0.3 M of KOH (Fig. 7), it can be seen that there are a lot of build-ups of meso and micropores in the structure.

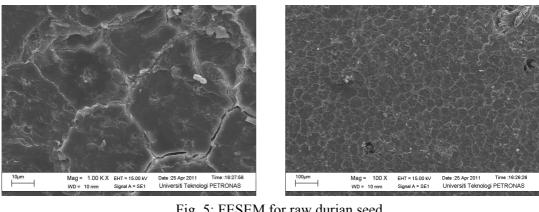


Fig. 5: FESEM for raw durian seed

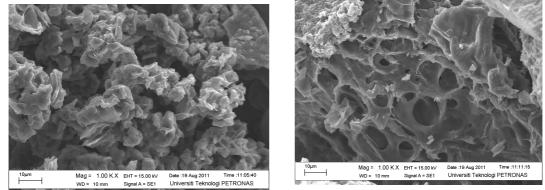


Fig. 6: FESEM for DSeAC – 0.60 M of KOH, 600°C for 1 hour

Fig. 7: FESEM for DSeAC - 0.30 M of KOH, 600°C for 2.5 hours

Fig. 7 shows the FESEM image result of dried durian seed at 600°C, for 2.5 hours and at 0.30 M of KOH. The structures have a lot of pores as compare to Fig. 6 FESEM results of dried durian seed at 600°C, for 1 hour and at 0.6 M of KOH. There is also a build-up of pore at the surface of the durian seed activated at 600°C, for 1 hour and at 0.60 M of KOH. This shows that there is a positive sign of meso and micropores formation in the structure.

Based on the FESEM micrograph it is proved that some macropores are created on the external surface of the carbon during the activation process. This phenomenon is mainly due to the release of volatile matter and reaction between potassium hydroxide and the carbon atom in the

precursor [9]. The presence of metallic potassium then intercalates the carbon matrix resulting in the widening of spaces between carbon atomic layers [10]. However, the increasing of the activation temperature would contribute to formation of new pores because more volatile organic compounds are released from precursor [11]. This is because longer heating duration would cause some of the pores become larger or even collapse, which leads to the reduction of surface area [12]. The increase of heating duration proved to have enhanced the development of micropores and total volume because the longer duration provides the possibility for generation of new micropores and mesopores in the carbon.

4. Conclusion

Based on the results that were obtained during this study, it was proven that durian shell and seed are the promising activated carbon precursors. The weight of yield product and characterization of the pore structure was influenced by the activation temperature and concentration of activating agent. The KOH concentration at 0.6 M and activation temperature of 400°C for 4 hours was found as the optimum conditions to acquire the highest yield and wider the micropores distribution. However, more study on the utilization of durian shell and seed as activated carbon biomass adsorbent can be done in future.

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