COMPARATIVE STUDIES ON DIFFERENT KIND OF DURIAN HUSKS AS MICROSTRUCTURE ACTIVATED CARBON

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In this study, chemical activation technique was used to produce the microstructure activated carbon (MAC) with particle size of 2000 micrometer using three types of durian husks. The durian husks used were *Durian D-2* (*DD-2*), *Durian Kampung* (*DK*) and *Durian KacingBaju* (*DKB*). The prepared MAC was characterized using proximate analysis and elemental analysis. The proximate analysis includes moisture content, ash content, volatile matter and fixed carbon. Besides that, iodine titration method was used to compare the absorbent properties of MAC with the silica gel as commercial absorbent. Absorption results showed that MAC (87.50%) possessed better absorbent ability as compared to silica gel which was only 52.08% for 80 minutes. The results indicated that all types of durian husk showed different values in physical analysis and almost same characteristics in chemical analysis.

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1. Introduction

Durian is a tropical fruit of trees species of Malvales order in Bombacae family and *genus* of *Durio*[1].In South East Asia countries durian (*Duriozibethinus)* is the most popular seasonal fruit [2].It is usually called as 'King of Fruits' which is a valuable tropical fruit in some Asian countries especially in Malaysia. Among the thirty known species of Durian, nine of them have been identified as producing edible fruits: *D. zibethinus*, *D. dulcis*, *D. grandiflorus*, *D. graveolens*, *D. kutejensis*, *D. lowianus*, *D. macrantha*, *D. oxleyanus* and *D. Testudinarum* [3].

According to the statistical data reported by the Ministry of Agricultural and Agro- Based Industry Malaysia, the local production of durian in the year 2011 was 300,470 metric tone, and about 255, 353 metric tone of it is the durian husk which was produced as the byproducts [4]. Therefore a proper way of managing these durian husks should be done in order to reduce the waste produced. One of the ways of reducing the waste produced by durian husk is by utilizing it to produce a product which would be beneficial. According to Tham, *et al*., these durian husks have the potential to be an active carbon [2].

Microstructure activated carbon (MAC) is also known as a solid, porous black carbonaceous material and tasteless [5]. The MAC obtained from agricultural byproducts has the advantages of offering an effective low cost replacement for non-renewable coal based granular activated carbon (GACs) provided that they have similar or better adsorption efficiency [6]. The

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MACs are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater [7].

Microstructure carbon can be classified that the microspores can be subdivided into two overlapping microporous regions such as ultra-microspores (with effective pore radii less than 0.7 nm), and super-microspores (having radii of 0.7 to 2 nm). Generally the microporous structure of an adsorbent is characterized by adsorption of gases and vapors and, to a small extent, by smallanglex-ray technique [8].

The aim of this study is the characterization of durian husk as MAC compound from three types of local durian which are *Durian Kacing Baju* (*DKB*), *Durian Kampung* (*DK*) as scientific name is *Duriozibesthinus Murray*, and *Durian D-2* (*DD-2*) with the local name of '*Dato Nina'*.

2. Materials and Methods

2.1 Fresh sample collection

The durian husk was obtained from Pasar Tani Jeli, Kelantan. The types of durian used were *Durian Kacing Baju* (*DKB*), *Durian Kampung (DK*) and *Durian D-2* (*DD-2*). These durians were ripe and the colours were greenish yellow with very strong smell and each weights around 4 to 5kg. The durian husk was washed before cut into small pieces range from 1 to 3 cm particle size [9].

2.2 Preparation of active carbon

The sample was then dried in the oven at temperature of 105 ˚C to 110 ˚C for 24 hours. It was then carbonized at the temperature of 450˚C for 1 hour then followed by activation. The preparation of MAC from durian husk was performed by chemical activation where potassium hydroxide (KOH) was used [10]. 25 g of dried durian husk was mixed with 100 ml of KOH solution, and then stirred. Then it was heated. The dried sample then transferred to an evaporating dish which was placed in a furnace and heated at 780˚C for 60 minutes. The MAC products washed sequentially with 0.5M HCI solution. Subsequently, the samples were repeatedly washed with hot distilled water until the pH of the solution reached from 5 to 8 and finally washed with cold distilled water. This is to remove residual organic and mineral matters. After that, the samples were dried for 24 hours and stored in desiccators [11].

2.3 Elemental analysis and chemical composition analysis

Elemental Analysis was done using energy dispersive X-ray technique (EDX) 7000/8000. Proximate analysis was done by analysing the moisture content, ash content, volatile matter and fixed carbon. The morphology and pore structure observed using scanning electron microscope (SEM) [15]. Following formula was used to determine fixed carbon as stated by Sugumaran *et al*. [9]:

$$Fixed\ carbon = 100 - (Ash\ content + Volatile\ matter) \quad (1)$$

2.4 Determination of the amount of iodine adsorbed

Different weight of MAC samples (0.2 to1.0 g) taken in a 250 mL flask and 10 mL of 5% HCI was added. The flask swirled until the carbon became wet. Then 100 mL of stock iodine solution (2.7 g of Iodine (Merck) and 4.1 g of potassium iodide (Merck) in 1 L of de-ionized water) were added to it and the mixture was shaken for 5 minutes in an orbital shaker and the samples were filtered. 50 mL of filtrate titrated with 0.1 M sodium thiosulphate until the solution become pale yellow. Then 1 mL of starch indicator solution (1%) was added and titration continued with sodium thiosulphate until the solution became colourless. A blank prepared without adding carbon [12, 14].

2.5 Statistical Analysis

Adsorption of iodine number conducted at varying adsorbent weight and contact time determined by Response Surface Design (RSD) using Statistical Analysis Software. There are total 13 runs of experiment provided by the design based on the maximum and minimum value of adsorbent weight and contact time. The minimum weight used was 0.1g and the maximum was 1.0g. The minimum time was 20 minutes and the maximum time was 80 minutes [13, 16].

3. Results and Discussion

3.1 Fresh sample collection

All the collected samples were dried to constant weight of 6% of moisture content. The samples were placed in desiccator before the preparation of MAC. There are 72% of weight changes before and after carbonization of the raw materials.

3.2 Preparation of active carbon

Table 1 shows the changes of amount of samples from raw material till it's sieved for granular size. There is 72% of change before carbonization and after carbonization. There are not much difference before and after activation.

Table 1: Average changes of weight of Durian Kacing Baju (DKB), Durian Kampung (DK) and Durian D-2 (DD-2).

Weight Change (g)			
Before carbonization	**After carbonization**	**After activation**	**After sieve**
65.91	22.10	22.06	8.78

Table 2: Proximate analysis of Durian Kacing Baju (DKB), Durian Kampung (DK) and Durian D-2 (DD-2).

Sample	**Raw material (%)**			**MAC (%)**			
	Moisture	**Ash**	**Volatile**	**Moisture**	**Ash**	**Volatile**	**Fixed Carbon**
DKB	14.3	5.3	2.3	8.7	35.0	9.7	86.8
DK	11.7	7.3	3.0	10.3	12.0	9.0	56.0
DD-2	12.3	5.3	3.7	5.6	15.7	14.3	70.0

Table 2 shows that *DK* has the highest moisture content, while *DD-2* has the lowest moisture content. It also shows the moisture content of raw materials of durian husk before turn into carbon. Compared to moisture content of raw material and active carbon of durian husk the moisture content of raw durian husk is higher. During active carbon preparation most of moisture content is reduced so the moisture content of active carbon is lower. It can be also observed that *DK* has the highest ash content while *DKB* has the lowest ash content. As for ash content of raw material *DK* has the highest ash content and the lowest is *DD-2.*

Ash content of MAC for durian husk is higher than raw materials. For the active carbon *DK* has better ash content compared to other two types since lower ash content indicates better active carbon it is. As for the volatile matter of the active carbon *DD-2* has the highest volatile

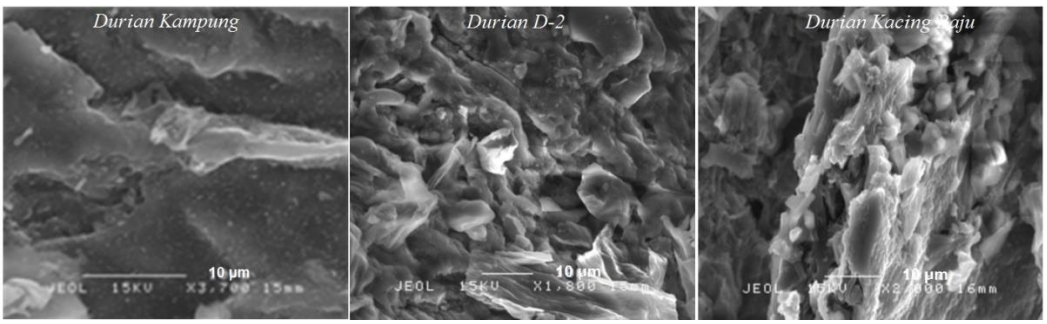
matter and *DK* has the lowest volatile matter. It means *DK* has better adsorption property compared to *DD-2* because the high content volatile matters will block the pores of adsorbent.

As for the raw samples, all the volatile matters are almost the same. If compared with raw durian samples. MAC raw samples have lower volatile matters because active carbon is highly amenable to adsorbing various volatile matters. It is because of its extremely high surface area and adsorption capacity, which eventually block the pores. The fixed carbon content of the coal is the carbon found in the material which is left after volatile materials are driven off. *DKB* has the highest fixed carbon and the lowest is *DK*.

3.3 Morphology and elemental analysis

From the Figure 1, it shows that SEM photograph of all samples are the same with wide variety of pores is present in MAC a long with fibrous structure. Carbon particles showed cavities, pores and more rough surfaces on the carbon samples. The pore size was about 2 to 3 cm.

The Table 3 illustrates the result energy dispersive weight of different types of element present. As shown in Table 2, among all the three types of durians the carbon *DKB* has the highest weight of carbon which is 92.6%. *DD-2* has the lowest weight of carbon which is 67.7%. *DKB* also have 4.8% of oxygen and 0.03% of silica. As for *DK* the amount oxygen present was 18.7% followed by silica 1.3%. For *DD-2* the amount of oxygen is 31.96% and silica is 0.30%.



(a) (b) (c)

Figure 1: SEM images of different types of active carbon at accelerating voltage of 15.00kV: (a) Durian Kampung, (b) Durian D-2, and (c) Durian Kacng Baju.

Table 3: Weight of different types of element present in activated Durian Kacing Baju (DKB), Durian Kampung (DK) and Durian D-2 (DD-2) obtained using Energy Dispersive X-Ray.

Element	% Weight		
	DKB	***DK***	***DD-2***
Carbon	92.63	80.07	67.74
Oxygen	4.77	18.67	31.96
Magnesium	0.30	-	-
Phosphorus	0.24	-	-
Silica	0.03	1.26	0.30
Potassium	1.83	-	-
Calcium	0.20	-	-
Total	100	100	100

3.4 Amount of iodine adsorbed

From the Table 5, it can be observed that different types of durian has different amount of iodine adsorbed. It can be observed that *DD-2* is better in adsorbing iodine than other samples. Since different types of weight were used it can be observed that 1.00 g is better at adsorbing

iodine. The optimum operating time is 80 minutes. The best adsorption of iodine is at 8 minutes and 1.00 gram and the reading is 87.5%. Both *DKB* and *DD-2* has the same reading.

Table 4: ANOVA Analysis for Durian KacingBaju (DKB), Durian Kampung (DK) and DD2.

N0	Source	Pr>F (DKB)	Pr>F (DK)	Pr>F (DD-2)
1	Time	0.0282	0.036608	0.0001
2	Weight	0.0001	0.0001	0.0001
3	Time* Weight	0.12147	0.349024	0.170642

3.5 Comparison with commercial adsorbent

The best result that obtained from adsorption is 80 minutes and 1.0 gram of active carbon used. Then it was compared with commercial adsorbent silica. The best reading for iodine adsorption for active carbon is 87.50% while for silica the reading was 52.08% for 80 minutes at 1 gram. From the results, it shows that the active carbon produced is better than commercial adsorbent used in this study.

Table 5: Amount of iodine adsorbed at 3 different time and weight with 13 runs using active carbon produced from Durian Kacing Baju (DKB), Durian Kampung (DK) and Durian D-2 (DD-2).

Weight (g)	Time (min)	Amount of iodine adsorbed (%)		
		DKB	***DK***	***DD-2***
0.1	20	0	0	0
0.55		59.38	21.88	46.88
1.00		71.88	71.88	65.63
0.1	50	6.25	3.13	9.38
0.55		37.50	56.25	37.5
0.55		37.50	53.13	37.5
0.55		40.63	53.13	40.63
0.55		40.63	53.13	40.63
0.55		37.50	56.25	40.63
1.0		78.13	78.13	68.75
0.1	80	30.18	28.13	34.38
0.55		59.38	75.00	71.88
1.00		87.50	84.38	87.50

4. Conclusion

In conclusion, three MAC made from different types of durian husk were characterized according to proximate analysis and chemical property, and compared. For proximate analysis like moisture ash and volatile matter showed differences among the three types of durians. As for chemical analysis like EDX the properties were similar. Absorption results showed that active carbon (87.50%) possessed better absorbent ability as compared to silica which was only 52.08% for 80 minutes. Therefore, the input from this study could initiate further investigation on this characterization of durian husk as MAC.

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References

[1] A. E. Putun, N. Ozbay, E. P. Onal, Putun E. Fuel Process Tech. **86**, 1207 (2005).
[2] Y. J. Tham, S. D. Arumugam, N. H. A. Latip, A. M. Abdullah, P. A. Latif. Environ. Asia. **3**,143 (2010).
[3] A. P Terzyk. Physiochemical. Eng.Aspects. **177**, 23 (2001).
[4] B. P. Shah, T. Pathak , S. Sankar, B. Suresh. Int. J. Pharm. Sci. Res. **5**(12), 878 (2014).
[5] D. Savova, E. Apak, E. Ekinci, F. Yardim, N. Petrova, T. Budinova.Biomass Bioenergy. **21**, 133 (2001).
[6] K. Tadvildari, G. Bigdeli, S. N. Eshafani, M. Farschi.J. Applies. Chem. Res. **11**, 47 (2009).
[7] K. Y. Foo, B. H. Hameed. Chem.Eng. J.**187**, 53 (2012).
[8] M.Danish, R. Hashim, M. N. Ibrahim, M. Rafatullah, T. Ahmad, O. Sulaiman. Bioresour. Tech. **6**(3), 3019 (2011).
[9] P. Sugumaran, S. V. Priya, P. Ravichandran, S. Seshadri. J. Sustainable. Energy. Environ. **3**, 125 (2012).
[10] R.F. Baseri, P.N. Palanisamy, P. Sivakumar. Pelagia Res.Library.**3**, 377 (2012).
[11] S. A. Mohammed, W. A. Najib, V. Muniandi. Int. J. Civ. Environ. Eng. **12**, 51 (2012).
[12] T.C. Chandra, M.M. Mirna, J. Sudaryanto, S.Ismadji. Chem. Eng. J. **127**, 121 (2007).
[13] T. Zhang, W. P. Walawender, L. T. Fan, M. Fan, D. Daugaard R. C. Brown. Chem. Eng. J. **105**, 53 (2004).
[14] Y. J. Tham, A. L. Puziah, A. M. Abdullah, D. A. Shamala, Y. H. Taufiq.Bioresour. Tech. **102**, 724 (2011).
[15] Y. Y. Voon, N. Sheikh Abdul Hamid, G. Rusul, A. Osman, S. Y. Quek. Food Chem. **103,** 217 (2007).
[16] Z. Qodah, R. Shawabkah. Brazilian J. Chem. Eng. **26**, 127 (2009).