INTRODUCTION

Activated carbon is porosity (space) enclosed by carbon atoms. It is an absorbent widely been applied in liquid or gas-phase application, as catalyst support, in food and beverage industries, pharmaceutical sectors and others due to the high porous texture and large adsorption capacity. The adsorption capacities of activated carbon depend not only on the surface area but other properties such as internal surface area, pore volume and pore size distribution also play important role as well\(^1,2\). Earlier, activated carbon derived from mangosteen peel was prepared by impregnating the sample with different phosphoric acid concentration (10, 20, 30 and 50 %) for 24 h with heating rate of 5 min together with activation duration for 20 min until the desired temperature 850 °C with presence of flushing of nitrogen gas. The sample was characterized by using micrometric ASAP 2010. The results revealed that acid concentration affect the surface area, yield of activated carbon and pore structures. The highest surface area with high micropore volume was obtained at 30 % acid concentration with 731 m\(^2\)g\(^{-1}\) and 0.33 cm\(^3\)g\(^{-1}\) respectively.

Key Words: Mangosteen husk, Phosphoric acid, Activated carbon, BET surface area.

Activated carbon can be prepared by employing physical or chemical activation or both. Physical activation involves carbonization of sample followed by activation of obtained chars in the presence of activating agents such as air, stream or carbon dioxide\(^6,11\) meanwhile chemical activation involve impregnation of material with certain chemical reagents within certain duration followed by carbonization\(^6\). Chemical activation offer some advantages if compared to physical activation. It gives higher yield and usually can be carried out at lower temperature\(^1,11,12\). Chemical agents that commonly used are zinc chloride, potassium hydroxide, sodium hydroxide, phosphoric acid and others\(^13\). However, phosphoric acid is preferred compared to zinc chloride due to environmental contamination\(^14\).

Mangosteen is one of the famous fruits in Malaysia and it is widely grown in Asian tropics. The weight of each fruits is approximately 55-75 g and only 25-30 % is considered as edible portion\(^15\). In Malaysia, approximately 29495 metric tones of mangosteen are generated in 2009 compared in 2003 which are only 18747 metric tones\(^16\). The fruit is gaining highly recognize recently by the grower and exporter. Furthermore, the rind or peel of the fruit posses hardened characteristics when exposed to air which make it as alternative candidate for activated carbon production.

Currently, there is no adsorption studies being conducted using mangosteen peel as adsorbents for activated carbon production. Therefore, this research is focusing mainly on assessed of mangosteen peel as absorbent and effect on...
impregnation ratio of phosphoric acid in the development of porosity in activated carbon preparation.

**EXPERIMENTAL**

**Sample preparation:** Mangosteen peels used in this study were collected from Serdang supermarket. The pulps were removed from the fruits and peels were washed several times with distilled water to remove dirt, colour and impurities present in the material. Then, the peel were cut to obtain particle size of 1-2 cm and dried at 70 °C for 2 h to reduce the moisture content. The clean draw mango peels was ensured to be completely dried to prevent formation of spores in the sample and was stored in desiccators for later use.

**Raw material characterization**

**Ultimate, proximate and biopolymer analysis:** The ultimate analysis consist of carbon, hydrogen, nitrogen, sulfur and oxygen was done by using Leco CHNS-932 elemental analyzer (Model Leco-Truspec®) while proximate analysis consist of ash and moisture were determined by using AOAC method\(^1\). Moisture content was determine by drying sample in oven at 135 °C for 2 h and ash by heating sample in muffle furnace at 600 °C for 2 h. Biopolymer analysis of cellulose, hemicellulose and lignin were analyzed by using fiber technology system 2010 instrument based on Van-Soest method.

**Thermogravimetric analysis:** The thermogram of raw mango peel was obtained using Mettler-Toledo (TGA/STDA 851e) instrument to determine the decomposition behaviour of raw material. Approximately, 12.4930 mg of sample were placed in ceramic crucible and heated from 25 to 1000 °C at a heating rate of 5 °C per min under flushing nitrogen gas (50 mL/min).

**Preparation of activated carbon using chemical activation:** Ten gram of mango peel was weighed and impregnated with 100 mL of different phosphoric acid concentration for 24 h in order to obtain different impregnation ratio. The impregnation ratio was calculated by assuming all the acid present in 100 mL of solution was taken up by the precursor. The impregnation ratio was expressed as gram of phosphorus per gram of dry precursor. By assuming that all the acid was well incorporated into the raw material, the impregnation ratio Xp’s was calculated to be 1:1.4, 1:2.8, 1:4.2 and 1:8.4 for acid concentration of 10, 20, 30 and 50 % respectively.

The wet impregnated samples were dried in an oven for 12 h at 85 °C and they were placed on a ceramic boat, which were then placed in the centre of a tube furnace. The furnace used in this study is programmable temperature controllers, which provided control of heating rate, temperature and heating time. The samples were heated at 850 °C with heating rate of 5 °C per min and continuous nitrogen flow of 150 cm\(^3\) min\(^1\) were supplied throughout the activation process. The samples were held at final temperature for 20 min before cooling down to room temperature. Then, the samples were washed with distilled water several times followed by 0.1 M NaOH solution until the pH of the filtrate reached approximately around pH 6-7. The wet samples were dried at 85 °C in oven overnight and weighed.

The yield of activated carbon is defined as the ratio of the weight of activated carbon to the original material with both weights on a dry basis.

**Activated carbon characterization**

**Physical characterization of prepared activated carbon:** In the process of characterization of prepared activated carbon, the sample was degassed at 200 °C for few hours to eliminate impurities and dehydrate the sample. The specific surface area and pore size distribution of the prepared activated carbon were determined by using automatic Micromeritics ASAP 2010 analyzer. The specific surface area was calculated using (Brunauer, Emmett and Teller) BET method. Total pore volume was determined by estimating the amount of nitrogen adsorbed at a relative pressure.

**Scanning electron microscope:** The scanning electron micrograph was done to identify the surface morphology of raw and activated carbon by using JEOI JSM 6400. Activated carbons that have good quality based on the results from physical characterization were selected for SEM analysis. Samples were dried for few hours before sticking the sample onto the stub using double-sided tape and were coated with gold coating. The stub was place into the vacuum chamber of SEM instrument. The morphology scanning were performed using different magnifications to obtain clear images.

**RESULTS AND DISCUSSION**

**Raw material characterization:** The results of ultimate, proximate and biopolymer analysis of raw mangosteen peel is tabulated in Table-1. The ultimate analysis consist of carbon, hydrogen, oxygen, nitrogen and sulfur are C-39.1 %, H-5.5 %, N-1.0 %, O-54.37 %, S-0.03 %, respectively. The results of ultimate analysis are very similar to activated carbon prepared from durian shell with C-39.3 %, H-5.9 %, N-1.0 %, O-53.74 %, S-0.06 %\(^1\). Proximate analysis gave 22.8 % of moisture and 9.9 % of ash conclusively. The high carbon content and reasonable ash content together with low inorganic matter derived from mangosteen peel revealed the potential of using this precursor since that is the important criteria in selecting material for activated carbon production. The differences of elemental analysis of this material compared to other material that successfully converted to activated carbon are given in Table-2. The chemical composition of mangosteen peel consist of cellulose, hemicellulose and lignin are 6.61, 12.15 and 48.63 % respectively. The fraction of lignocellulosic composition in the precursor mainly influences the pore distribution in activated carbon produced. In addition, the porosity development in activated carbon mainly depends on the nature of precursor together with method employed for activated carbon production\(^1\). Ncibi et al.\(^1\), stated that the presence of high lignin content derived from the raw precursor can be considered as capable precursor in activated carbon production. Apart from that, the hardness posses by mango peel after exposure to air indicate the suitable of precursor in production of granular activated carbon.

**Thermogravimetric analysis:** Fig. 1 represent the thermogravimetric (TG) and differential thermogravimetric (DTG) curve of the raw mangosteen peel conducted under
The decomposition reaction of pyrolysis in the material can be articulated by following reaction:\(^\text{2}\):

\[
\text{Biomass} \rightarrow \text{solid residue} + \text{volatiles}
\]

The thermal decomposition of the mangosteen peel occurs in four stages. The first stage occurred at 80 ºC, second at 270 ºC, third around 320 ºC and fourth and finally at 700 ºC. The first stage might due moisture released from the material with mass loss (22 %). The second stage and third stage correspond to hemicelluloses and cellulose degradation which occur around 200-400 ºC\(^3\). As seen in Fig. 1, the last stage involves degradation of lignin, which occurs throughout the whole range of temperature\(^2\). In addition, difficulty arise in comparison of thermal degradation of one material to another due to different chemical composition (cellulose, hemicelluloses and lignin fraction), heating rate, temperature, inorganic substances present in the sample are the factor affecting the thermal behaviour\(^2\).

**Activated carbon characterization**

**Effect of acid concentration on yield of activated carbon:** The yield of activated carbon prepared at different phosphoric acid concentration is displayed in Fig. 2 in term of percentage. The yield of activated carbon can be elaborated as ratio of the weight of resultant activated carbon to the original precursor, mangosteen. The yield of activated carbon prepared at different phosphoric acid concentration is in range of 30.73-35.09 %. The pattern showed that the activated carbon yield was decrease from 32.18 to 30.73 % when the ratio of phosphoric acid increased from 10 to 20 % concentration. However, at higher phosphoric acid concentration 30-50 %, the yield was observed to increase. Chemical activation commonly increase the yield of activated carbon and phosphoric acid as activating agent play important role during carbonization by promotes depolymerization, dehydration and redistribution of chemical composition followed by conversion of aliphatic compounds to aromatic compounds and thereby enhances the yield of activated carbon\(^1\).

![Fig. 2. Yield of activated carbon obtained acid concentration. The error bar represent the mean ± standard error where n = 3](image)

**Effect of acid concentration on pore development:** Impregnation ratio is one of the important variables in controlling the frame of pore networks. A series of activated carbon with different phosphoric acid concentration were prepared and evaluated in order to study the effect of impregnation ratio. The pore characteristic obtained for all the prepared activated carbon was tabulated in Table-3. As shown in Table-3, initially the surface area of raw mangosteen peel was found to be 0.66 m\(^2\) g\(^{-1}\) only, which did not even exceed 1 m\(^2\) g\(^{-1}\). After carbonization, the raw material impregnated with different concentrations of phosphoric acid, all the activated carbons produced

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**TABLE-1**

ULTIMATE, PROXIMATE AND BIOPOLYMER ANALYSIS OF RAW MANGOSTEEN PEEL (MEAN ± SE, n = 3)

<table>
<thead>
<tr>
<th>Component</th>
<th>Value (± SE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>39.1 ± 4.30</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>5.50 ± 0.30</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>1.00 ± 0.20</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.03 ± 0.02</td>
</tr>
<tr>
<td>Oxygen*</td>
<td>54.37</td>
</tr>
<tr>
<td>Moisture b</td>
<td>22.8</td>
</tr>
<tr>
<td>Ash</td>
<td>9.90</td>
</tr>
<tr>
<td>Volatile matter</td>
<td>12.9</td>
</tr>
<tr>
<td>Fixed Carbon*</td>
<td>54.4</td>
</tr>
<tr>
<td>Biopolymer analysis* (%)</td>
<td></td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>12.15</td>
</tr>
<tr>
<td>Cellulose</td>
<td>6.61</td>
</tr>
<tr>
<td>Lignin</td>
<td>48.63</td>
</tr>
</tbody>
</table>

*a*Estimated by differences, *b*values on dry basic, *c*values on wet basic

**TABLE-2**

COMPARISON OF ELEMENTAL ANALYSIS OF THIS RESEARCH WITH OTHER STUDIES

<table>
<thead>
<tr>
<th>Description</th>
<th>Ref. 12</th>
<th>Ref. 1</th>
<th>Ref. 7</th>
<th>This work</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw material</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbon content</td>
<td>35</td>
<td>59.31</td>
<td>49.6</td>
<td>39.1</td>
</tr>
<tr>
<td>Oxygen content</td>
<td>-</td>
<td>28.74</td>
<td>38.2</td>
<td>54.37</td>
</tr>
<tr>
<td>Ash content</td>
<td>4</td>
<td>0.30</td>
<td>2.60</td>
<td>9.90</td>
</tr>
</tbody>
</table>

**Phosphoric acid concentration**

![Fig. 1. Thermogravimetric/differential thermogravimetric curve of raw mangosteen peel](image)
showed significant changes and extensively produce higher surface area compared to the raw precursor. This proved that chemical activation offer advantages by creating larger surface area. In the context of surface area, the low impregnation ratio 1:1.4 or 10% acid concentration, the surface area is 143 m$^2$/g, which is not in the range of activated carbon synthesized that generally used in industry. Basically, the range of activated carbon was between 700-1500 m$^2$/g, which is not in the range of activated carbon synthesized that are microporous solid. An absence of hysteresis loop indicates the absence of mesoporosity and can suggest that the carbon prepared consist mostly of micropores with only small portion of mesopores, which can be concluded that porosity remain restricted in micropore which occurred at high impregnation ratio$^{24}$. However, the enhancement of nitrogen uptake can be observed as phosphoric acid concentration increased. As seen in Fig. 4, the total pore volume and micropore volume increased in correlation with acid concentration. In term of ratio of micropore volume to total volume ($V_{mic}/V_{tot}$) for 10, 20, 30 and 50% are 1.00, 1.07, 1.06 and 1.13, respectively.

Conclusively, based on cost consumption and chemical recovery, the optimum acid concentration selected was 30%. The acid concentration based on highest surface area ($731$ m$^2$/g), micropore volume (0.33 cm$^3$/g) and total volume (0.35 cm$^3$/g).

**Examination of scanning electron microscope:** The surface morphology of raw mangosteen peel and selected activated carbon prepared with acid treatment was observed using scanning electron microscope. Fig. 5 illustrates the morphology of raw precursor. The surface of raw precursor is hard and poorly developed. In addition, the evidence for the material is poorly developed can be assigned through the poor or negligible BET surface area (Table-3). Compare to raw precursor, the activated carbon prepared with acid activation followed by carbonization and activation shows completely

### TABLE-3

<table>
<thead>
<tr>
<th>Sample (%)</th>
<th>$S_{BET}$(m$^2$/g)</th>
<th>Micropore surface area (m$^2$/g)</th>
<th>Micropore volume (cm$^3$/g)</th>
<th>Total pore volume (cm$^3$/g)</th>
<th>Average pore diameter (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw material</td>
<td>0.6598</td>
<td>-0.19</td>
<td>-0.000113</td>
<td>0.00014</td>
<td>3.68</td>
</tr>
<tr>
<td>AC-10</td>
<td>143</td>
<td>125</td>
<td>0.07</td>
<td>0.07</td>
<td>9.91</td>
</tr>
<tr>
<td>AC-20</td>
<td>667</td>
<td>564</td>
<td>0.30</td>
<td>0.32</td>
<td>9.62</td>
</tr>
<tr>
<td>AC-30</td>
<td>731</td>
<td>627</td>
<td>0.33</td>
<td>0.35</td>
<td>9.76</td>
</tr>
<tr>
<td>AC-50</td>
<td>756</td>
<td>569</td>
<td>0.30</td>
<td>0.34</td>
<td>9.18</td>
</tr>
</tbody>
</table>

**Fig. 3.** Distribution of pore volume in activated carbon assessed at different acid concentration

**Fig. 4.** Adsorption/desorption isotherms of activated carbon assessed at different acid concentration

**Fig. 5.** SEM images of raw material at different magnification
smooth and clear appearances (Fig. 6). The heat treatment during carbonization creates formation of pores in the activated carbon by removing all the volatile compounds and impurities present in the material. Apart from that, washing process with sodium hydroxide is important in activated carbon production since it involves in creation of pores and makes the pores accessible.

**Conclusion**

Mangosteen peel can be used as precursor for activated carbon preparation due to presence of high carbon content and reasonable ash content. The carbonization/heat treatment of raw material creates formation of pores in the sample. In correspond to thermogram, the suitable activation temperature for preparation of activated carbon is higher than 700 °C. All the activated carbon prepared with different phosphoric acid from mangosteen consists of micropores. The 30 % acid concentrations is the most suitable for activated carbon production. However, further studies by employing different parameters need to be carried out to assess the optimum condition for activated carbon production.

**ACKNOWLEDGEMENTS**

The authors would like to express sincere thanks to Ministry of Science, Technology and Innovation, Malaysia for financial support this research under Science Fund (06-01-04-SF0677).

**REFERENCES**