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Waste Palm Shell Converted to High Efficient Activated Carbon by Chemical Activation Method and Its Adsorption Capacity Tested by Water Filtration

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Abstract

The concentrations of phosphoric acid on the characteristics of high efficient activated carbon were investigated. Adsorptions capacities of activated carbon prepared by chemical method were carried out to evaluate the adsorptive of some heavy metals. It was found that treated (palm shell by sulphuric acid, 5\%) raw material reduces contain of impurities and increases Brunet Elmer Teller (BET) surface area of the prepared activated carbon. The most advantageous BET surface area was obtained with following conditions; phosphoric acid concentration was 30\%, treated raw material and 500\,^\circ\text{C} was activation temperature with holding time of 2 hours. Obtained BET surface area is 1058 m\textsuperscript{2}\,g\textsuperscript{-1} and the average pore diameter was found 20.64 nm. Scanning electron microscopy (SEM) also confirmed the pore size distribution on the activated carbon. The maximum thermal stability was observed up to 600\,^\circ\text{C} by thermal gravimetric analyzer (TGA). Adsorption capacity of the activated carbon was tested passed by artificially polluted water through a water filtering column and it was observed 100\% of Cr was adsorbed followed by Pb (99.8 \%), Cd (99.5 \%) and Cu (25 \%).

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Keywords: treated palm shell, high pure activated carbon; chemical method; adsorbing capacity; filtering system

1. Introduction
Substances with high carbon content such as coal, wood, and coconut shell can be used in the production of activated carbon. The raw material has a very large influence on the characteristics and performance of activated carbon. Activation refers to the development of the adsorption properties of carbon. Raw materials such as coal and charcoal do have some adsorption capacity, but this is greatly enhanced by activation process.

Activated carbon has the strongest physical adsorption forces of the highest volume of adsorbing porosity of any material known to mankind. It is a black, solid substance resembling granular or powdered charcoal and extremely porous with a very large surface area. Its surface area can reach up to more than 1000 m²/g. In other words, five grams of activated carbon can have the surface area of a football field.

There are three main forms of activated carbon: i) granular Activated Carbon (GAC); Irregular shaped particles sizes ranging from 0.2 to 5.0 mm. This type of activated carbon is used in both liquid and gas phase applications, ii) powder Activated Carbon (PAC); Pulverized carbon with a size predominantly less than 0.18 mm (US mess 80). These are mainly used in liquid phase applications and for flue gas treatment, iii) pellet Activated Carbon; extruded and cylindrical shaped with diameters from 0.8 to 5 mm. They are mainly used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content.

In general, characteristics of activated carbon are controlled by the manufacturing process. Depending on the nature of the raw materials, the nature of activating agent and the condition of the activation process, the properties of activated carbon can be varied. The surface of activated carbon can contain protonated (C-OH₂ +), neutral (COH) or ionized (CO-) groups. Activated carbon with protonated surfaces are also known as H-type carbons while activated carbons with ionized surfaces are known as L–type carbons [2].

2. Materials & Method

The Palm shell was obtained from Felda Chalok Barat mill, Setiu, Terengganu, Malaysia. It was used as the pioneer for the preparation of activated carbon. The shell was crushed and sieved to different particle sizes, namely, less than 2.0 mm. The palm shell particles were soaked with 5% acid solution to loosen the fiber and traces. This is an important idea to make sure the activated carbon that would be produced of good quality. The raw palm shell were immersed in the solution for 24 hours. After 24 hours the waste floated at the surface of acid solution and decanted. Then the palm shell particles were washed by the distilled water until acid was removed.

2.1. Carbonization of palm shell

Precursor, shell particles, were taken the weight around 20g. The precursor was later sunk with around 100 mL of freshly prepared concentrated solution of H₃PO₄ with different concentration percentages: 10%, 20% and 30% in the clay. The samples were carbonized for 2 hours. The temperature was raised from room temperature to 650°C with a heating rate of 10°C/min. After the retention time 2 hours, the samples were cooled down for another 2 hours. Then the activated carbons were soaked with distilled water until the pH was around 5.0. The smaller values of pH affect the surface area of the activated carbon. Lowest values of pH show that the surfaces. Then SEM instrument was used to determine the pore size and the morphology of the surface area of activated carbon. Brunauer, Emmett and Teller (BET) method [3] was used to measure the surface area of the activated carbon. Analysis was done using a Micromeritic Quantachrome at Malaysia Institute Nuclear Technology (MINT), Bangi, Selangor. About 0.2-0.3 g of the prepared activated carbon was put in the sample tube and left to constant heating at 250°C
until the pressure was stable at 6x10^-6 torr. N₂ gas adsorption at 77 K was used in this analysis. Surface area was determined by using the BET equation, calculated from the isotherms, by assuming that the area value of a nitrogen gas molecule to be 16.2 Å.

3. Results and Discussion

3.1. The phosphoric acid concentration on the yield of activated carbon

The overall yields of activated carbons prepared at five different concentrations of phosphoric acid are shown in Figure 1. Activated carbons used in this part of the study were prepared by impregnating palm shells with different concentration of phosphoric acid and then activated at 500°C for 2 hours.

Increasing the concentration of phosphoric acid from 10% to 20% increases the yield of activated carbon; the initial effect of phosphoric acid is to inhibit the release of volatile matter, which results in a higher yield of activated carbon. Similar trends were also reported by Rodriguez-Reinosa and Molina Sabio [4] and Guo and Lua [5] in their studies on the preparation of activated carbons from lignocellulosic materials and palm shell respectively by chemical activation. Subsequently, further increases in the percentages resulting continual decreases in the yield of activated carbon. From Figure 1, it can be seen that a further increase in phosphoric acid concentration, releases the volatiles and therefore decreases the yield of activated carbon.

Fig. 1. The effect of the concentration of phosphoric acid on the yield of activated carbon prepared from palm shell

In this case, the weight loss of shell is caused by volatile lumps. In the carbonization process, tar is probably the predominant product of devolatilization indicated by a significant weight loss (60% w/w) in temperature range 400 to 500°C. Scanning electron microscope was used to study surface morphology
and pore size of the untreated and treated samples. Figures 2 (a, b) show the surface of treated and untreated activated carbon for retention time of 2 hours, \(\text{H}_3\text{PO}_4\) concentration of 10% to 30% and pyrolysis temperature at 500\(^\circ\text{C}\). It can be seen from Figure 4 (a, b) smooth surfaces with many orderly pores were developed. Comparing all the Figures, Figure 4(b) shows the highest development of pore, the development of pore was due to the effect of \(\text{H}_3\text{PO}_4\) concentration. Increasing the acid concentration accompanied the development of pore. This is due to the roles of impregnating agent that minimizes the formation of tars and other liquid, which could clog up the pores and inhibit the development of pores structures.

When the concentration of \(\text{H}_3\text{PO}_4\) increases, the development of pore also increases (see Figure 2 (b)) but with some components attracted to the surface. The components probably come from the untreated raw material. Thus they react with the surface of the raw material during carbonization and affect the pore distribution of activated carbon. Hence, it produces a roughly smooth surface with lack development of pores. Surface area analysis of activated carbon by palm shell was performed using a Micromeritic Quantachrome by nitrogen gas adsorption at 77k. The surface area plot of untreated and treatment of activated carbon with impregnated with various concentration of phosphoric acid is given in Figure 3. Both curves that BET surface area increases rapidly with increasing of phosphoric acid concentration.

![Surface Area vs. Phosphoric Acid Solution](image)

**Fig. 3.** The surface area of treated and untreated activated carbon

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Average Pore Diameter(Å)</th>
<th>Total Pore Volume (cc/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>U10</td>
<td>2.137E+01</td>
<td>1.961E-01</td>
</tr>
<tr>
<td>U20</td>
<td>2.126E+01</td>
<td>2.129E-01</td>
</tr>
<tr>
<td>U30</td>
<td>2.063E+01</td>
<td>3.611E-01</td>
</tr>
<tr>
<td>T10</td>
<td>2.147E+01</td>
<td>2.445E-01</td>
</tr>
<tr>
<td>T20</td>
<td>2.143E+01</td>
<td>3.758E-01</td>
</tr>
<tr>
<td>T30</td>
<td>2.064E+01</td>
<td>5.457E-01</td>
</tr>
</tbody>
</table>

Table 1. Effect of untreated, treated activated carbon on pore diameter and pore volume with different phosphoric concentration.

The adsorptive capacity of adsorbent is related to its internal surface and pore volume. This case, treated activated carbons show the optimum surface area, 1058 m\(^2\)g\(^{-1}\). Meanwhile, the optimum surface area obtained from untreated activated carbon was 700.1 m\(^2\)g\(^{-1}\). This result shows that the treatment of raw material of activated carbon increases the development of activated carbon surface area and increases the adsorptive capacity of activated carbon. Treatment the raw material of adsorbent were loose fiber and traces, therefore it reduces interference during the carbonization process. Besides surface area, pore diameter is also an important characteristic of the activated carbon. The decrease in pore diameter
increases the total pore volume of activated carbon. In this study, the result of average pore diameter and total pore volume of activated carbon are shown in Table 1. The results showed that untreated samples contribute to smaller pore volume compared to treated samples. This case, the maximum result of pore volume was achieved at T30 (5.457E-01 cc/g). Different phosphoric acid concentrations also affect the contributions of pore volume and pore diameter. The increase in the concentration of the treated and untreated samples increases the pore volume, and vice versa for the case of pore diameter.

3.2. The study of absorption capacity of activated carbon with artificial waste water (Cu, Pb, Cd, Cr) on filter column design

The study on adsorption capacity using artificial waste water is shows in Table 2. In this study four (Pb, Cu, Cr, Cd) elements of heavy metals were studied. The results showed that activated carbon column gives highest adsorption for Cr element but rather poor (25%) for Cu. In the case of Cd and Pb, the result shows around 99% of elements were absorbed in the activated carbon filter column. Basically, adsorption is a mass transfer process by which a substance is transferred from liquid phase to a surface solid, and then bounded by physical and/or chemical interaction [8]. Probably, the higher absorption capacity of Cd, Pb and Cr is caused by the tendency of interaction of elements on the surface of activated carbon.

### Table 2. Obtained result from application of activated carbon in filter column

<table>
<thead>
<tr>
<th>Elements</th>
<th>Pb µgL⁻¹</th>
<th>Cu µgL⁻¹</th>
<th>Cr µgL⁻¹</th>
<th>Cd µgL⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conc. of elements in water before filtration</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Conc. of element filtrate water after filtration</td>
<td>0.204±2.2</td>
<td>75.404±1.4</td>
<td>NA</td>
<td>0.455±1.0</td>
</tr>
</tbody>
</table>

4. Conclusion

Activated carbon was prepared from palm shells by chemical activation in H₃PO₄ maintained at 500°C. The quality of activated carbon is highly dependent on the preparations condition. For this study, the optimum condition for preparing microporous activated carbon with high pore surface area and pore volume were the treatment with 20% H₂SO₄ in solution at 24 hours and sank in 30% H₃PO₄ in solution. In this study, the thermal stability of activated carbon is important. It was observed that the optimum thermal stability of activated carbon was around 600°C. Adsorption studies for activated carbon impregnated with 30% H₃PO₄ in the application of filter system confirm the adsorbing capacity of activated carbon. This activated carbon has good adsorbing capacities for cadmium, chromium, and lead but quite poor for copper. In conclusion, the prepared activated carbon is good for removal of toxic elements in water.

References
