

## Production of Activated Carbon from Palm Oil Shell Waste and Its Adsorption Characteristics

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### Abstract

The effects of raw material treatment and concentration of phosphoric acid on the characteristics of activated carbon were investigated. Adsorptions of Cu, Pb, Cr and Cd in activated carbon were carried out to evaluate the adsorptive capacity of selected activated carbons. It was found that treated raw material reduces inorganic element and increases Brunet Elmer Teller (BET) surface area of the activated carbon. The optimum BET surface area was achieved with following conditions; 30% phosphoric acid concentration, treated raw material, and activation temperature of 500°C with holding time of 2 hours. The optimum BET surface area and the average pore diameter were found to be 1058 m<sup>2</sup>g<sup>-1</sup> and 20.64nm, respectively. The pore size distribution of the activated carbon by scanning electron microscopy (SEM) also confirmed to these conditions. Thermal stability of these samples was investigated using thermal gravimetric analyzer (TGA). The maximum thermal stability was observed up to 600°C. Adsorption studies for activated carbon impregnated with 30% H<sub>3</sub>PO<sub>4</sub> in the application of filter system confirm the adsorbing capacity of activated carbon. In this study, it was observed 100% of Cr was adsorbed followed by Pb (99.8 %), Cd (99.5 %) and Cu (25 %).

*Keywords: Activated carbon; phosphoric acid; absorbing capacity; filter system; microporosity.*

### 1.0 Introduction

Activated carbon is a crude material from graphite. One of the applications of this substance is as pencil lead. Activated carbon differs from graphite by having the random imperfect structure, which is highly porous over a broad range of pore size from visible cracks and crevices to molecular dimensions. The graphite structure gives the carbon a very large surface area, which allows the carbon to adsorb a wide range of compounds.

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 m<sup>2</sup>/g. In other words, five grams of activated carbon can have the surface area of football field.

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Substances with high carbon content such 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.

There are three main forms of activated carbon:

- Granular Activated Carbon (GAC)
  - Irregular shaped particles sizes ranging from 0.2 to 5mm. This type of activated carbon is used in both liquid and gas phase applications.
- 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.
- Pellet Activated Carbon
  - Extruded and cylindrical shaped with diameters from 0.8 to 5mm. They are mainly used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content.

Activated carbon consists mainly of carbon (87 to 97%) and other elements such as hydrogen, oxygen, sulfur and nitrogen. Various compounds are also present either originating from the material or generated during its preparation. Activated carbon also can adsorb various substances both from gas and liquid phases. This ability justifies it as an adsorbent [1].

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 ( $\text{C-OH}_2^+$ ), neutral (COH) or ionized ( $\text{CO}^-$ ) 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].

Certain contaminants accumulated on the surface of the activated carbon. This is called adsorption. The two main reasons why chemicals adsorb onto activated carbon are:

- a “dislike” of the water
- attraction to the activated carbon.

Adsorption of most contaminants results from both reasons. There are many organic compounds, such as chlorinated and non chlorinated solvents, gasoline, pesticides and trihalomethanes. Activated carbon is also effective for removal of some of chlorine and moderately effective for removal of some heavy metals [2].

## **2.0 Materials & Method**

Palm shell was used as the precursor for the preparation of activated carbon. The raw material was obtained from Felda Chalok Barat mill, Setiu, Terengganu, Malaysia. The shell was crushed and sieved to different particle sizes, namely, less than 2.0 mm.

### 2.1 *Pretreatment of material*

The raw materials were soaked with 10% acid solution to loose the fiber and traces. This is the important part to make sure the activated carbons that would be produced are of good quality. The raw materials were immersed in the solution for 24 hours. After 24 hours the waste floated at the surface of acid solution. Then the wastes were separated from the material by the use of distilled water.

### 2.2 *Carbonization of palm shell*

Precursor, palm shell, was taken the weight around 20g. The precursor was later sunk with around 100 ml of freshly prepared concentrated solution of H<sub>3</sub>PO<sub>4</sub> with different concentration percentages: 10%, 15%, 20%, 25% and 30% in the clay.

One step activation was performed in an electrical furnace. The samples were carbonized for 2 hours. The temperature was raised from room temperature to 1230°C with a heating rate of 10°C/min. After the retention time, the samples were cooled down for around 2 hours. Then the activated carbons were soaked with distilled water until the pH was around 4.5. The measurements of pH of activated carbon are important. The smaller values of pH affect the surface area of the activated carbon. Lowest values of pH show that the surfaces of activated carbon have some unwanted element that interfere the pore of activated carbon.

Thermogravimetric analysis was performed using Perkin Elmer Pyris 6 TGA at a heating rate of 25°C/min. Sample of approximately 100mg was heated from 30°C to 800°C. The instrument was purged with nitrogen flow at a rate of 20.00 ml/min.

SEM instrument was used to determine the pore size and the morphology of the surface area of activated carbon. The analysis was carried out at the Institute of Oceanography (INOS), KUSTEM by using JEOL JSM-6360LA with Energy Dissemination Analyses X-ray equipment (EDAX) and the result was recorded using Kodax Verichrome Pan VP 120. A small amount of sample was placed on top of a stub, which has a diameter of about 10 mm and was then coated with gold by using JEOL JFC -1600 auto fine coating.

### 2.3 *Determination of surface area (BET Method)*

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  $6 \times 10^{-6}$  torr. N<sub>2</sub> 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.

### 3.0 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.

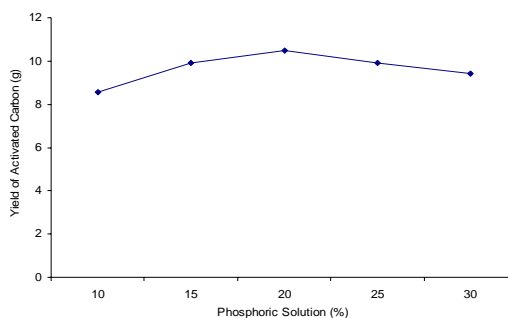


Figure 1 The effect of the concentration of phosphoric acid on the yield of activated carbon prepared from palm shell

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 Rodriquez -Reinosa and Molina Sabio [4] and Guo and Lua [5] in their studies on the preparation of activated carbons from lignocellulosic materials and oil-palm shell respectively by chemical activation. Subsequently, further increases in the percentages resulting continual decreases in the yield of activated carbon. For these impregnated samples the phosphoric acid assumes a dehydration agent role during activation. It inhibits the formation of tars and any other liquids that can clog up the pores of the samples. With phosphoric acid impregnation, the movement of volatiles through the pore passages will not be hindered and volatiles will be released from the carbon surface during activation. 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.

#### 3.2 Temperature effect on activated carbon preparation

The temperature effect on the prepared activated carbon was investigated using thermogravimetric analysis (TGA). The TGA profiles obtained under inert gas condition are presented in Figures 2 and 3.

The first weight losses around 100°C are observed for the samples. The decrease in weight loss is probably caused by thermodesorption of physically adsorbed material such as water vapor, hydrocarbon [6], and in the case of raw material, residual volatile content. For the raw material, the same trend was also observed when the temperature reaches 380°C.

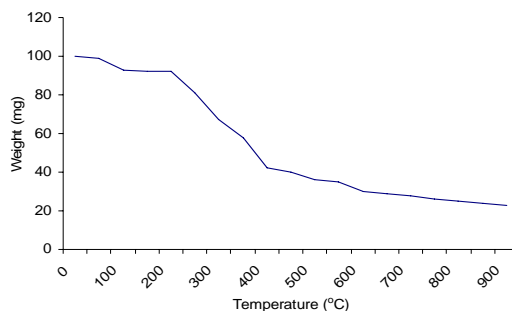


Figure 2 Thermal stability of raw material (palm shell)

At a higher temperature, the raw material samples present a gradual and pronounced weight loss up to 900°C. 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.

In contrast, the treated activated carbon samples do not present any significant weight loss until the temperature reaches 600°C. Probably the volatile content of raw material was released during carbonization process.

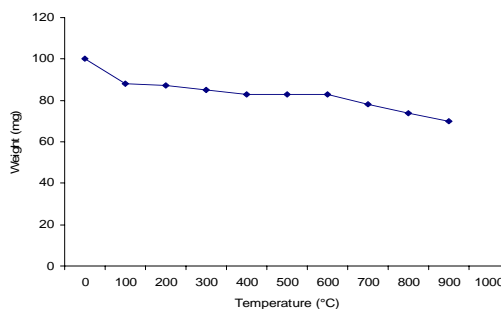


Figure 3 Thermal stability of activated carbon treated with 30% H<sub>3</sub>PO<sub>4</sub>

### 3.3 Scanning emission microscopy morphology of prepared activated carbon

Scanning electron microscope was used to study surface morphology and pore size of the untreated and treated samples. Figures 4.4 (a,b) and 4.5 (a,b) show the surface of treated and untreated activated carbon for retention time of 2 hours, H<sub>3</sub>PO<sub>4</sub> concentration of 10%, 30% and pyrolysis temperature at 500°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 H<sub>3</sub>PO<sub>4</sub> 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.

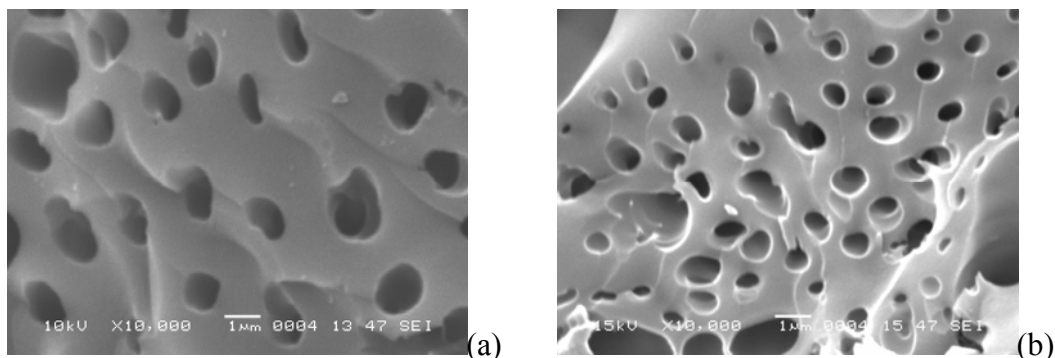


Figure 4 SEM Photographs of activated carbon porosity after treatment at different phosphoric acid solution (a) 10% phosphoric acid solution (b) 30% phosphoric acid solution

Figure 5(a) shows that some components interfere with the surface of activated carbon and reduce the development of pores. This is because the 10% concentration of phosphoric is not enough to minimize the formation of tars and other components that affect the development of pores.

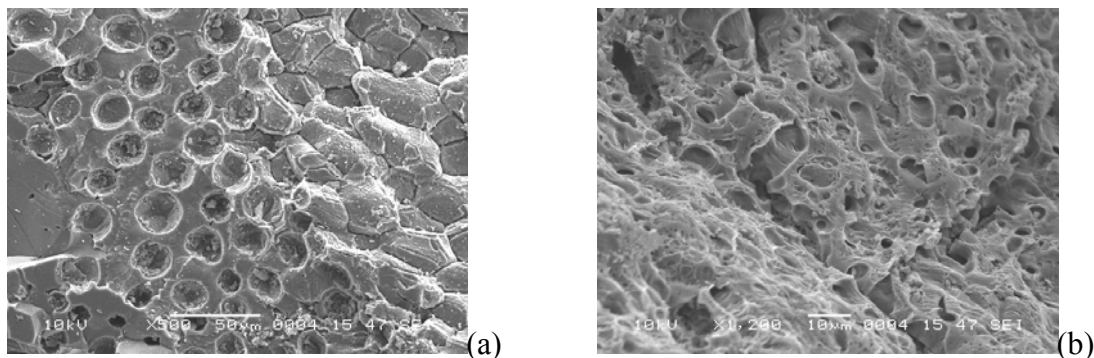


Figure 5 SEM Photographs of activated carbon porosity before treatment at different phosphoric acid solution (a) 10% phosphoric acid solution (c) 30% phosphoric acid solution

When the concentration of  $H_2PO_3$  increases, the development of pore also increases (see Figure 5 (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.

### 3.3 Surface area analysis

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 6. Both curves that BET surface area increases rapidly with increasing of phosphoric acid concentration.

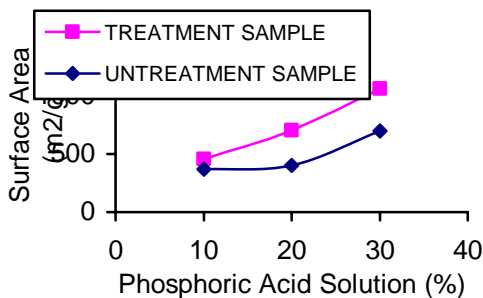


Figure 6 The surface area of treated and untreated activated carbon

The adsorptive capacity of adsorbent is related to its internal surface and pore volume. Generally, the larger the specific surface area of the adsorbent, the better its adsorptive performance will be [7]. In this case, treated activated carbons show the optimum surface area, 1058 m<sup>2</sup>g<sup>-1</sup>. Meanwhile, the optimum surface area obtained from untreated activated carbon was 700.1 m<sup>2</sup>g<sup>-1</sup>.

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. Hence, the developments of pore were increased.

### 3.4 Pore diameter and pore volume analysis

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. Hence, it increases the distribution of surface area.

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. In 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.

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

Sample ID	Average Pore Diameter Å	Total Pore Volume (cc/g)
U10	2.137E+01	1.961E-01
U30	2.063E+01	3.611E-01
T10	2.147E+01	2.445E-01
T30	2.064E+01	5.457E-01

### 3.5 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 shown 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.

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

Elements	Pb $\mu\text{gL}^{-1}$	Cu $\mu\text{gL}^{-1}$	Cr $\mu\text{gL}^{-1}$	Cd $\mu\text{gL}^{-1}$
Concentration of elements before filtration	100	100	100	100
Concentration of element after filtration	0.204 $\pm$ 2.2	75.404 $\pm$ 1.4	NA	0.455 $\pm$ 1.0

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.

## 4.0 Conclusion

Activated carbon was prepared from palm shells by chemical activation in  $\text{H}_3\text{PO}_4$  maintained at  $500^\circ\text{C}$ . The quality of activated carbon is highly dependent on the preparation 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%  $\text{H}_2\text{SO}_4$  in solution at 24 hours and sank in 30%  $\text{H}_2\text{PO}_4$  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  $600^\circ\text{C}$ .

Adsorption studies for activated carbon impregnated with 30%  $\text{H}_3\text{PO}_4$  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.

## Acknowledgements

We greatly acknowledge the financial support from KUSTEM through vot 54045 and 54196. Assistance from Mr. Asrul, Tarmizi, Jamal, Pn Asbah and Miss Zuliani is also acknowledged.

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