# ACTIVATED CARBON FROM PALM SHELL, AN AGRICULTURAL WASTE, USING $K_2CO_3$: INFLUENCE OF ACTIVATION TIME ON PORE DEVELOPMENT

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## ABSTRACT

The main objective in this work is study the influence of activation times in the preparation of activated carbon by the method of potassium carbonate ($K_2CO_3$)-chemical activation. The characteristics and pore development of the prepared activated carbon were investigated. The experiments were carried out with activation time varied from 0.5 to 4.0 h. The results show that in all cases, increasing the activation time the yield decreases, while the adsorption of $CO_2$ will increase, progressively. It was found that the specific surface area of activated was at a maximum value (about 1170 $m^2/g$) at a activation time of 2.0 h with carbonization temperature of $800^0C$ and at an impregnation ratio of 1.0.

## INTRODUCTION

Activated carbon is one of the most important microporous adsorbents from an industrial view of point. Therefore it has been widely used for separation of gases, recovery of solvents, removal of organic pollutants from drinking water and a catalyst support. As environmental pollution is becoming a more serious problem, the need for activated carbon is growing. It is a versatile adsorbent because of its good adsorption properties. There are basically two methods for preparing activated carbon: physical and chemical activation. Physical activation consists of two steps: the carbonization of the starting material at high temperature followed by the activation of the char by using carbon dioxide or steam [1]. In chemical activation, the raw material is impregnated with an activating agent and heated in an inert atmosphere. The carbonization step and the activation step proceed simultaneously and pores are developed with dehydration and oxidation processes.

However, activated carbon prepared from palm shell using $K_2CO_3$ activation for carbonaceous precursors has not been thoroughly investigated. The aim of this work is to use $K_2CO_3$ as activating agent and study the influences of activation time on pore development and yield.

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## EXPERIMENTAL

Palm shell were mixed with saturated solution of chemical activation agent ($K_2CO_3$) and kneaded. This Mixture was then dried in an oven kept at $110^0C$ for 24 h to prepare the impregnated sample. In this wok, the impregnation ratio of 1.0 was used.

$$\text{impregnation ratio} = \frac{(\text{weight of } K_2CO_3 \text{ in solution})}{(\text{weight of palm shell})} \qquad (1)$$

Typical carbonization run began by changing 100g of impregnated sample in the reactor and heated up to the carbonization temperature in flowing stream of nitrogen (15 l/min). The temperature of reactor was increased at the rate of $10^0C$ /min, until it reached the final carbonization temperature. The activation times were varied from 0.5 to 4.0 h with carbonization temperature of $800^0C$, respectively. After activation time, the sample was cooled down under nitrogen ($N_2$) flow, and was washed sequentially several times with hot water, and finally with cold distilled water to remove residual. Then the sample was dried at $110^0C$.
Yield was calculated based on weight of Palm shell and chemical recovery was estimated using the following equation:

$$\text{Yield} = \frac{(\text{weight of palm shell before activation})}{(\text{weight of activated carbon produce})} \qquad (2)$$

$$\text{Chemical recovery} =$$
$$\frac{(\text{weight of sample before washing}) - (\text{weight of sample after washing})}{(\text{weight of impregnated chemical})} \qquad (3)$$

The characterization of activated carbon samples is carried out using $CO_2$ adsorption at 273 K using a Micromeritics ASAP 2010 surface area analyzer. Before the experiment began, the adsorbents were degassed ($10^{-4} mmHg$) at 393 K. The surface areas of the samples were measured based on Brunauer-Emmet-Teller (BET) method, the Dubinin-Radushkevich (DR) method report analyzed was applied to calculate the micropore volume. The pore size distribution (PSD) was obtained from the Horvath-Kawazoe (HK) analyzed. The solid density of activated carbon was measured by helium displacement with an ultrapycnometer (AccuPyc 1330 pycnometer).

## RESULTS AND DISCUSSION

Figure 1 shows the BET specific surface area of activated carbon at different activation time. The BET specific surface area increases in accordance to the activation time of 0.5 to 2.0 h and decreases slightly at the activation time of 2.0 to 4.0 h. The maximum specific area is obtained 1170 $m^2$/g. Therefore, it is deduced that $K_2CO_3$ works effectively as activation reagent below 2.0 h. This progressive activation time increases

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the C-$K_2CO_3$ reaction rate, resulting in increasing carbon "burn-off ''. Concurrently, the volatiles from the samples continue to evolve with increasing activation time. The devolatilization process further develops the rudimentary pore structure in the char, whereas the C-$K_2CO_3$ reaction enhances the existing pores and creates new porosities [2].
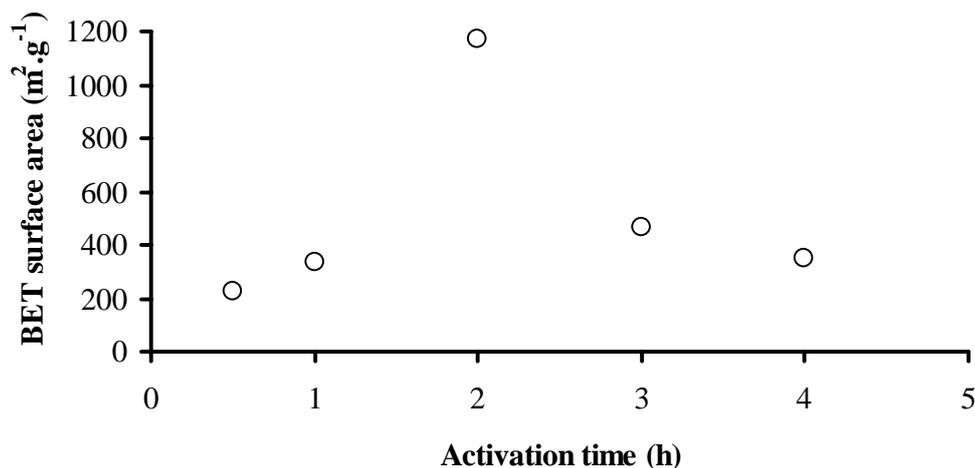


Figure 1: Influence of activation time on the BET specific surface area.

The typical adsorption isotherms for activated carbon produced from palm shell are shown in Figure 2. Within the influence of activation times investigated, Type I isotherms, the amount of $CO_2$ adsorbed at $P/P_o=0.03$ increased for activated carbons prepared at relatively longer activation time. At short activation time, one could clearly see that initial step region is abruptly followed by a plateau indicating that the adsorption has virtually stopped because multi layer of adsorbate can not be formed due to close proximity of the pore wall. It also implies the near absence of mesopore and macropore inside the material. Further increase in activation time through activation process has widened the pores as well as increased the volume of micro pore and total of pore volume.

Variations in the micropore with $K_2CO_3$ for sample prepared from activation time at 0.5 h to 4.0 h are shown in Figure 3. The micropore volume increases with activation time from 0.5 to 2.0 h and than between 2.0 to 4.0 h, the microvolume area slightly decreased, because surface area of the microporous were converted to mesopore volume.
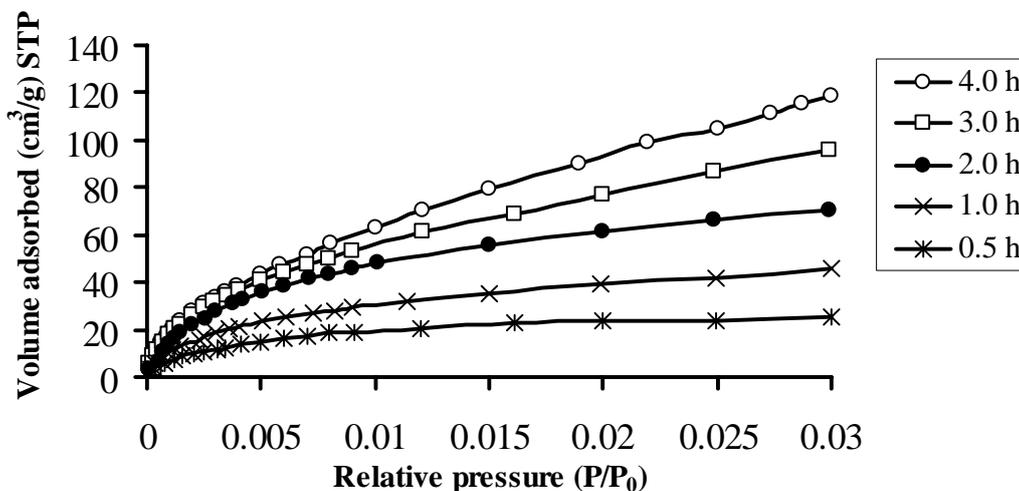
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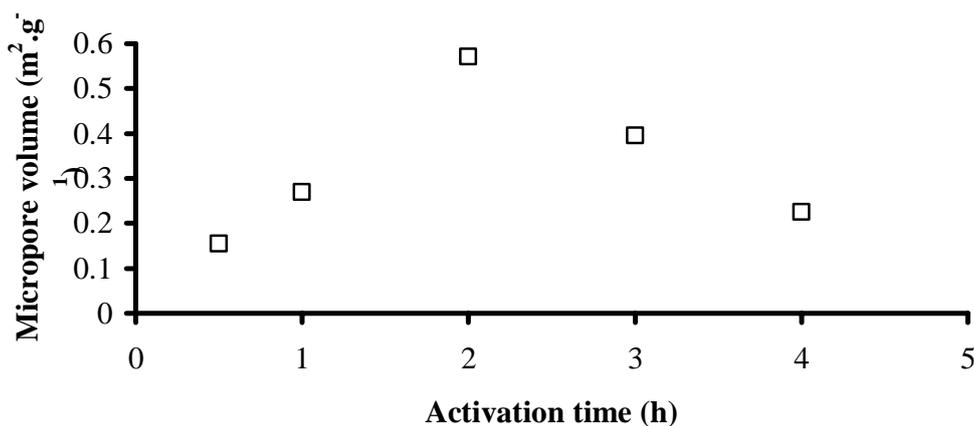Figure 2: $CO_2$ adsorption isotherms of activated carbon at different activation time.



Figure 3: Micropore volume of activated carbon at different activation time.

The influence of activation times on the pore development of activated carbon produced is very significant as seen in Figure 4. By increasing the activation time from 0.5 to 4.0 h increases the evolution of volatile matters from the precursor, leading to increase of the pore development, and creates new pores. At short activation time, the pore size of activated carbon produced mainly consist of micropore, however with increase of activation time, the creation of micropores to mesopore also increase [3].

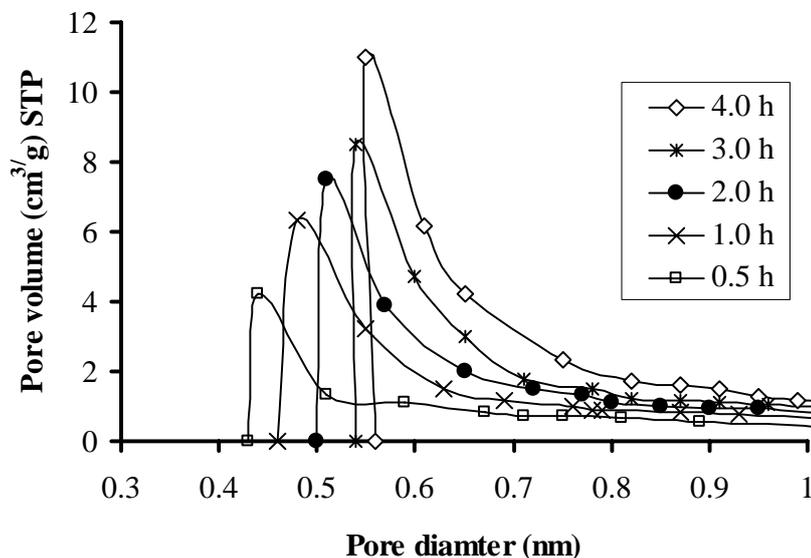Corresponding Author: nata_pm.yahoo.com

Figure 5: the influence of activation times in the pore development.

The solid density of activated carbon was 1.8568, 2.0584, 2.3421, 2.6210, and 2.7129 $g/cm^3$ for the activation time of 0.5, 1.0, 2.0, 3.0, and 4.0, respectively. Increasing the activation time increased the solid density progressively due to the release of low-molecular weight products (water, furan derivates and laevoglucose) as a result of dehydration and elimination reactions, leading to evolution of internal porosity [4]. Under the same condition when the activation time was 0.5, 1.0, 2.0, 3.0, and 4.0 the corresponding yield of activated carbon was 27.12, 23.35, 18.86, 17.67, and 13.73 %, respectively. How ever, weight losses during activation processes were de to totally different reaction mechanism or each activation method. When chemical activation with $K_2CO_3$ impregnation was used, as the activation time increasing release of volatile products as a result of intensifying dehydration and elimination reaction, it is also clear that char of the palm shell is gasified by $K_2CO_3$. McKee (1983) studied the gasification of graphite by alkali metal compounds and found that $K_2CO_3$ was reduced by carbon in an inert atmosphere as follows:

$$K_2CO_3 + 2C \longrightarrow 2K + 3CO$$
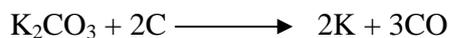
The recovery ratio decreases with an increase of activation time. This results support previous findings that $K_2CO_3$ is reduced by carbon in inert atmospheres, whereby palm shell char has reacted with $K_2CO_3$ and than $K_2CO_3$ was removed during gasification. There in this work, above 2.0 h, the carbon has reduced the impregnated $K_2CO_3$ and was consumed through the formation of CO. Thus the specific surface area and the pore volume increased.
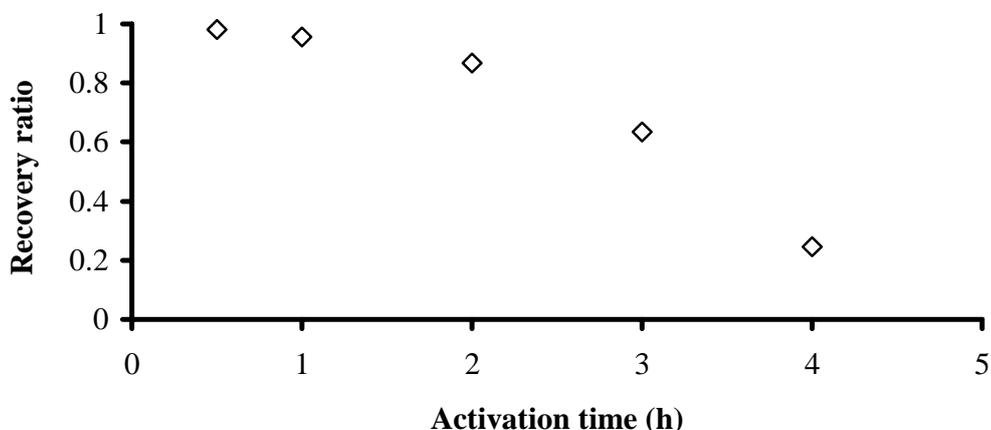
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Figure 5: the chemical recovery ratio at different activation time.

## CONCLUSION

The pore development in palm shell based activated carbon by chemical activation with $K_2CO_3$ depends strongly on activation time. The result was found that the specific surface area of activated was at a maximum value (about 1170 $m^2$/g) at a activation time of 2h with carbonization temperature of 800 $^0$C and at an impregnation ratio of 1.0. The increasing the activation time decreased yield, increased adsorption isotherm of $CO_2$ and increased solid density. From the results of yield of activated carbon and reagent recovery ratio, it was concluded that the carbon involved in the palm shell char was removed as CO by reduction of $K_2CO_3$ above 800$^0$C.

## ACKNOWLEDGMENTS

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