# Preparation and Characterization of Green Porous Palm Shell Based Activated Carbon by Two Step Chemical Activation Using KOH

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**Abstract.** The large quantity of agricultural waste materials that poses disposal challenge to our environment could be converted into useful products such as activated carbon (AC). Palm oil shell based porous AC was prepared by two step process using KOH as the chemical activant. Palm oil shell was carbonized at 800°C for 2 hours and activated using $CO_2$ at same temperature for 1 hour which yield 23.27% fixed carbon. The AC was characterized by Langmuir surface area, BET surface area and pore volume of 410.7 $m^2$/g, 350 $m^2$/g and 0.2 $cm^3$/g respectively, the FTIR analysis identified the presence of alkanes, carbonyls and hydroxyls as the main functional groups in the AC. Scanning electron microscopy images illustrates the gradual formation of pores from the precursor to the produced AC due to elimination of volatiles and contaminants in the material. However, the AC produced showed basic properties suitable for the removal of hydrophobic organic contaminants in water and wastewater.

## Introduction

The presence of agricultural waste poses disposal challenge to areas with rapid growth of agriculture. However, Palm oil is the leading oil crop cultivated in the Southeast Asian countries for the production of cooking oil. Malaysia as example, is the largest exporter of palm oil in the world with over 5 million hectares of cultivated lands at an average production rate of 19.69 ton/hectare of fresh fruit bunch. About 2 million tons were generated annually as waste from such industry and among these waste are palm oil shell [1, 2]. Converting such agricultural waste to useful materials such as adsorbents could solve their disposal problems. Commercially, the precursor materials used commonly for AC production is coal but due to its high cost, high demand and non-renewable nature made researchers to look for alternatives. Agricultural residues like palm oil shell are substitutes because they are readily available, cheap and possess suitable properties such as low inorganic materials content and high percentage composition of volatiles which helps to create the basic pore structure as they escape during carbonization processes [3].

The chemicals mostly used during activation are $K_2CO_3$, LiOH, $ZnCl_2$, $H_3PO_4$, NaOH and KOH [4-6]. Hence, the main aim of this study is to produce porous AC from palm oil shell using KOH as chemical activant with specific properties towards organic contaminants removal in water and wastewater.

## Experimental

Materials

Palm oil shell was obtained from a local factory in Parit Raja, Johor, Malaysia. The palm shells were first washed with deionized water several times and then dried at 105°C for 24hrs in hot

air oven. The palm shells were then crushed and separated using sieves and shaker into sizes between 0.6–1.18mm which were used as starting material and levelled as raw palm shell (RPS).

Sample preparation

The precursor was chemically activated after carbonizing it initially from ambient temperature to carbonization temperature of 800°C under nitrogen flow of $150cm^3$/min for 2hrs at 10°C/min heating rate. The carbonized sample was then impregnated with KOH in the ratio 1:2 and the mixture was loaded into the reactor for subsequent heating to a temperature of 800°C for 1hr at 10°C/min heating rate in the presence of nitrogen gas at $100cm^3$/min. Then it was heated for activation under $CO_2$ flow of $150cm^3$/min at 800°C (10°C/min) for 1hr. After activation, the samples were allowed to cool down to ambient temperature under nitrogen flow after which it was removed from the furnace. The carbon produced were then washed with 0.1M HCl, hot water and then by deionized water until the washing's pH were between 6-7 and it was dried in the oven at 105°C for 24hrs. The AC produced was labelled as potassium carbonate treated carbon (PHAC) [7].

Characterization

The synthesized AC was characterized by BET surface area using Micromeritics ASAP 2020 for full isotherms analysis. Scanning electron microscopy (SEM) model JOEL JSM-7600F was carried out for both the raw precursor RPS and the activated carbon PHAC produced to study the pore development and surface morphology of the materials. The surface functionalities on the materials were analysed by Fourier transform Infrared Spectroscopy (FTIR) spectra recorded between 4000 $cm^{-1}$ and 600 $cm^{-1}$ at 4 $cm^{-1}$ resolution and 16 scans $min^{-1}$ using Parking Elmer Spectrum-100 model.

**Results and Discussion**
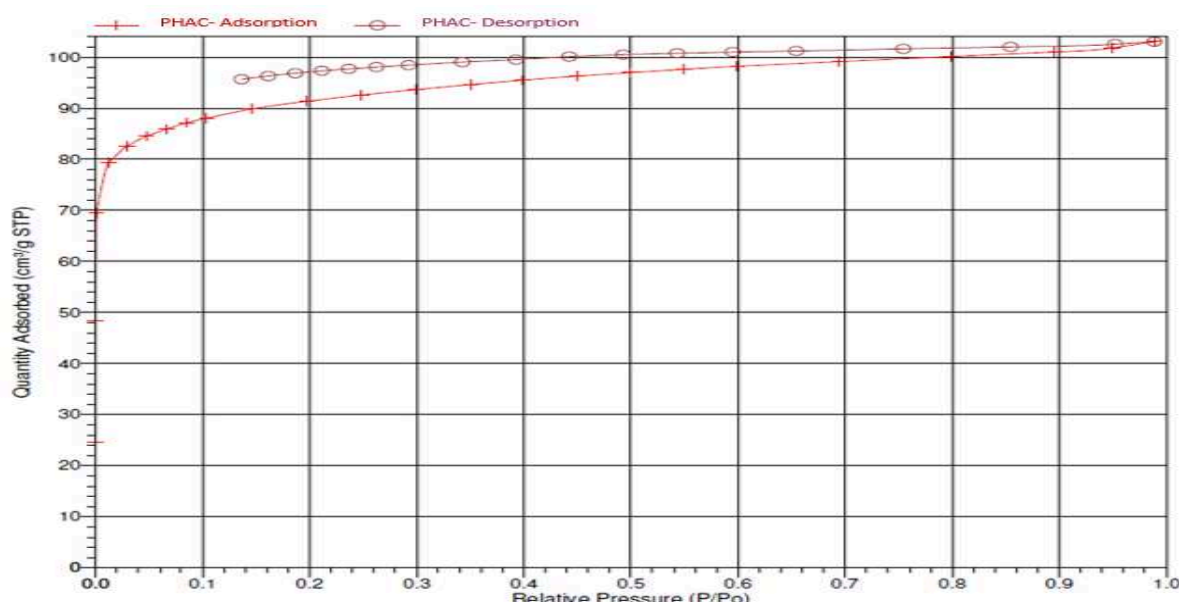
Surface area and pore volume



Figure. 1 Type I isotherm linear plot from $N_2$ adsorption analysis of PHAC

The adsorptive capacity of activated carbon is its most important property which in general is proportional to the surface area [2, 8]. The Langmuir surface area, BET surface area and pore volume of the activated carbon are 410.7 $m^2$/g, 350 $m^2$/g and 0.2 $cm^3$/g respectively. The results compares with the studies obtained by Daud and Ali [9], where 260 $m^2$/g from palm oil shell. They showed that, the specific surface area is achieved around 800°C and it decreases above 800-1000°C.

However, KOH is a good chemical activant around 800°C as increasing the carbonization temperature above 800°C increased the reaction rate between carbon and KOH which leads to increased carbon burn-off [10]. In fig. 2 above, it could be seen that the linear branch of the isotherms becomes curved which is no longer parallel to the pressure axis and a loop begin to appear which indicates a region of mesopores development [2].

Proximate analysis

Table 1. Proximate analysis of RPS and PHAC

| Sample | Moisture (%) | Volatiles (%) | Ash (%) | Fixed carbon (%) |
|---|---|---|---|---|
| RPS | 5.60 | 68.75 | 2.38 | 23.27 |
| PHAC | 4.50 | 7.40 | 7.50 | 80.60 |

The proximate analysis of both raw precursor and the AC were presented in Table 1 with PHAC having relatively high percentage of fixed carbon 80.60% which is due to high removal of volatiles from the precursor as indicated by higher percentage of volatiles in it (68.75%).

Elemental analysis

Table 2. Proximate and ultimate analysis showing percentage decomposition of palm oil shell in this study compared with other studies

| Proximate Analysis (%) | Daud and Ali, 2004 | Shin et al., 2014 | Khan et al., 2014 | This Study |
|---|---|---|---|---|
| Moisture | 7.96 | 4.0 | 9.61 | 5.60 |
| Volatiles | 72.47 | 63.0 | 80.92 | 68.75 |
| Ash | 1.10 | 5.0 | 4.31 | 2.38 |
| Fixed Carbon | 18.70 | 27.0 | 14.67 | 23.27 |
| **Ultimate Analysis (%)** | | | | |
| Carbon | 50.10 | 47.24 | 49.74 | 47.35 |
| Hydrogen | 6.85 | 4.87 | 5.68 | 5.40 |
| Nitrogen | 1.90 | 0.24 | 1.02 | 1.08 |
| Sulphur | - | 0.00 | 0.27 | 0.06 |
| Oxygen (by difference) | 41.15 | 47.65 | 43.36 | 46.11 |

The precursor material has low ash content as seen on Table 2 which is considered as an added advantage for carbon synthesis. The fixed carbon content in this study (23.27%) when compared with other studies, it is high as presented in Table 2 [9, 11, 12]. The high percentage composition of carbon in palm oil shell from the ultimate analysis (47.35%) is an indication of a good precursor material for production of activated carbon [2].
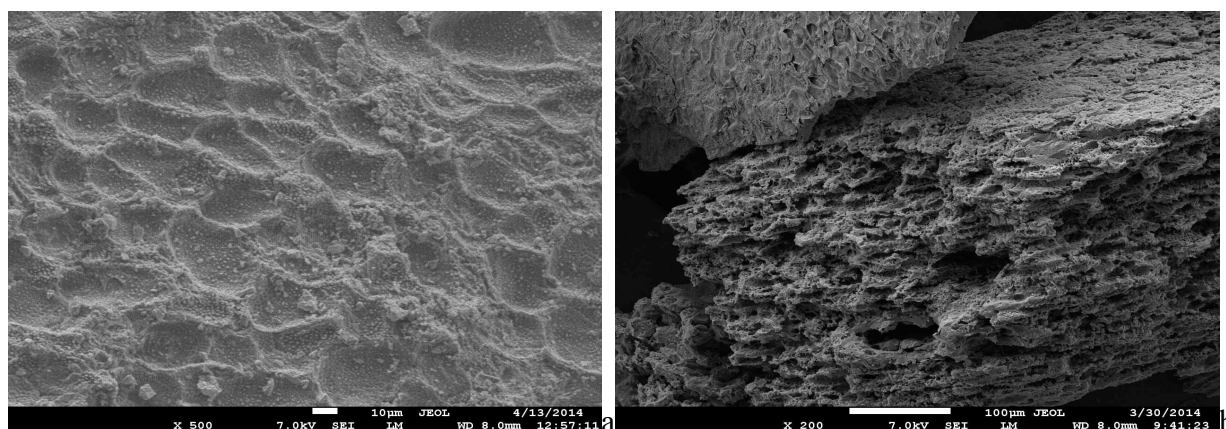
SEM



Figure 2. SEM images of RPS (a) and PHAC (b)

The SEM analysis of the RPS and PHAC were presented in Figure 2a-c. The surface morphology of the RPS showed no pores formation due to presence of volatiles and contaminants on its surface. However, there is development and widening of pores from the carbonized precursor to PHAC. This phenomena is more pronounced in PHAC as a result of the space created by loss of moisture, volatiles (hemicellulose, cellulose and lignin) and the chemical activant that remained in the cavities which is leached through washing with 0.1M HCl [13].
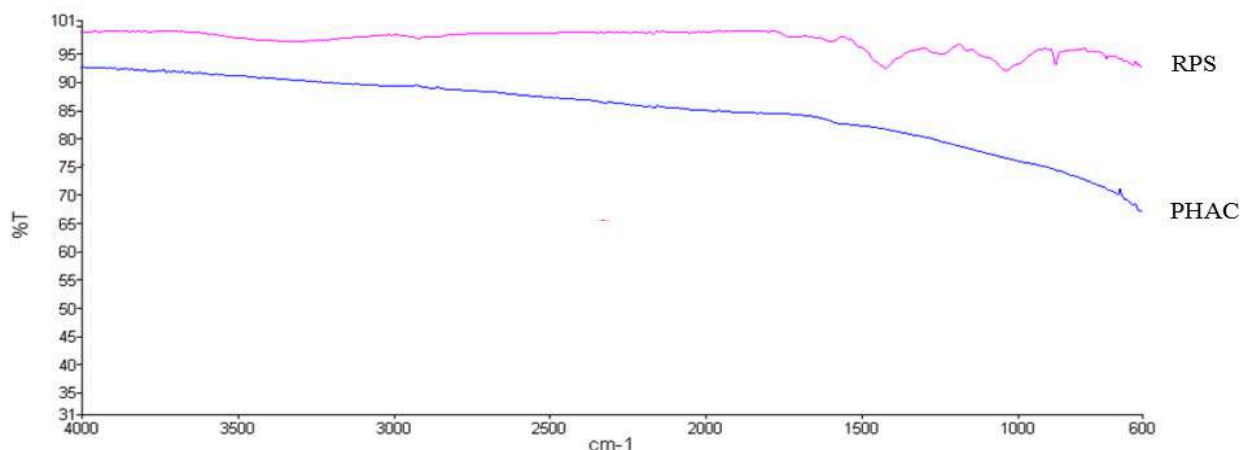
FTIR



Figure 3. FTIR spectra of RPS and PHAC

The spectra of the raw precursor (RPS) as in Figure 3, shows the bands at 621cm$^{-1}$, 875, 1033, 1594 and 3330 cm$^{-1}$ that corresponds to out of plane bending in benzene derivatives, C-O-C stretching vibration (in ester, ether and phenol), C-O stretching in ethers, C = C stretching in in aromatic rings and OH stretching vibration in OH functional groups respectively. However, the FTIR spectra of PHAC indicate elimination of some peaks at (875, 1594 and 3330 cm$^{-1}$) as result of decomposition of functional groups and release of volatiles through carbonization process. Also, peaks at 620 and 3882 cm$^{-1}$ were introduced, which corresponds to OH stretching vibration (in free alcohols and phenols) and bending vibration from carbon (triple bond) or C-H bond from alkynes [2].

## Conclusion

The activated carbon prepared from palm oil shell by chemical activation with KOH attained a maximum of 410.7 $m^2$/g, 350 $m^2$/g and 0.2 $cm^3$/g as Langmuir surface area, BET surface area and pore volume at carbonization temperature of 800°C with activation time of 2h and impregnation ratio of 1:2. The increase in carbonization temperature decreases the surface area while increase in impregnation ratio increase it. The activation process and type of activant employed allows for accurately controlling the pore size distribution of the prepared carbon. Moreover, the process leads to synthesis of adsorbent material with suitable properties for a specific contaminant removal in water and wastewater.

## Acknowledgment

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