

## Oxidised Biochar from Palm Kernel Shell for Eco-friendly Pollution Management

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### ABSTRACT

*Oil palm plantations produce palm kernel shell (PKS) that can be converted into biochar for environment-friendly soil remediation and water treatment. Oxidation with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) may enhance surface characteristics and the quality of low-rank PKS biochar as a sorbent for environmental decontamination. This study aims to determine the effect of oxidation on the surface characteristics (i.e., specific surface area, surface charge, and chemical properties) of PKS biochar, and compared with that of PKS activated carbon. The surface area for the oxidised PKS biochar was similar to that of PKS biochar, indicating that oxidation did not remove the pore blocking material from the surface area of the PKS biochar. However, oxidation has increased the amount of negatively charged oxygen functional groups in PKS biochar, as indicated by the analyses of the Fourier transform infrared spectroscopy (FTIR) and cation exchange capacity (CEC). The CEC value of raw and activated PKS biochar were similar and 4.6 and 2.6 times lower for PKS biochar and oxidised PKS biochar, respectively. Oxidation caused enlargement of pores on PKS biochar and caused a reduction of specific surface area. More research is required to establish the process conditions to create a greater surface area and sorption capacity.*

**Keywords:** *oxidised biochar, activated carbon, oxygen functional groups, pore enlargement, BET surface area, carbonisation, decarboxylation, decarboxylation*



## INTRODUCTION

The palm oil industry is producing edible oil and biodiesel to fulfil the global demand, contributing USD\$ 4500 million of income to Malaysia alone [1]. The continued worldwide increase in palm oil demand has resulted in deforestation and expansion into peatland, causing habitat and biodiversity loss [2] as well as carbon loss in the form of carbon dioxide (CO<sub>2</sub>) [3]. Intensive production of palm oil has also generated millions of tons of agricultural residues such as palm oil mill effluent, empty fruit bunches, oil palm fronds, mesocarp fibres and palm kernel shell (PKS) [4]. These unsustainable practices have resulted in numerous media campaigns against palm oil and the establishment of sustainability criteria under the assembly of the Roundtable for Sustainable Palm Oil (RSPO) [5]. The sustainability of the oil palm industry relies on two strategies: (1) intensification of the process to obtain more oil per hectare land [6], and (2) valorisation of biomass residues for reducing the impact of solid waste to the environment [7, 8]. Solid wastes from the plantation and milling process have been reused as mulch and fuel. The residual oil in PKS contributes to its high calorific value [9], which is partially reused as in-house fuel for boilers, or sold as fuel to conventional coal fire power plants [10]. The by-products of the combustion process (i.e., biochar) may be reused as a sorbent for decontamination of the environment.

An emerging value-added approach is the pyrolytic conversion of agricultural residues to biochar and activated carbon [11]. Granular activated carbon from coal has been used traditionally as an adsorbent in the management of pollutants present in industrial effluent [12] as well as soil and sediments [13]. Since coal is not a sustainable resource, substitution with biochar for water and soil treatment is a promising alternative [14]. Although physically activated carbon has been an effective sorbent for removing pollutants, the cost of producing activated carbon is higher than that of biochar [15] due to the requirement of high temperatures (i.e. >800°C) and use of superheated steam. Even though biochar is cheaper to produce, it is inferior to activated carbon in terms of porous structure [16]. Biochar has a lower capacity to remove pollutants than activated carbon due to its lower micropore volume and surface area. Further modification of PKS biochar with H<sub>2</sub>O<sub>2</sub> may improve sorption capacity through (1) the removal of pore-blocking tar compounds that enhance its pore structure,

and (2) introduction of more polar oxygen functional groups on the surface [17]. It has been reported that oxidised biochar produced from bamboo and peanut hull possessed a greater surface area and more functional groups than original biochar [18, 19]. Furthermore, the preparation of activated carbon usually involves usage of concentrated chemicals such as alkaline and acids that additional step to remove residuals from the activated carbon, resulting in additional cost in chemical and water consumption. Using  $H_2O_2$  for oxidising biochar is relatively clean as no residual is left with completion reaction. Therefore, post treatment using  $H_2O_2$  may be a cost effect way to improve value of PKS biochar as sorbent.

Although there have been reports on the synthesis of oxidised biochar with  $H_2O_2$ , the effect of oxidation on the surface and physico-chemical characteristics of PKS biochar has not been studied yet. This article elucidates the effects of  $H_2O_2$  oxidation reaction on the decontamination capacity of PKS biochar in terms of iodine number and BET ( $N_2$ ) surface area, chemical characteristics in terms of surface functional groups and CEC, and surface microstructure with scanning electron microscopy (SEM). The physico-chemical characteristics of PKS biochar were compared with those of PKS activated carbon. Thermogravimetric analysis was used to determine the moisture, volatile matter, fixed carbon and ash content of the materials.

## METHODOLOGY

### Preparation of PKS, PKS Activated Carbon, PKS Biochar, and Oxidised PKS Biochar

Raw PKS was collected from Endau Palm Oil Mill Sdn Bhd at Petaling Jaya, Selangor. The raw PKS was cleaned with distilled water to remove impurities, oven-dried at  $110^\circ C$  overnight, pulverised and sieved to obtained fine particles with diameters between 1-2 mm. For preparing PKS activated carbon, 1 g of PKS was mixed with 3 mL of 65%  $H_3PO_4$  and kept for 26 hrs at room temperature. Excess  $H_3PO_4$  was removed by thorough rinsing with distilled water until pH 7, oven-dried for 24 hrs, and pyrolysed in the furnace at  $500^\circ C$  for 1.5 hrs [20]. The PKS biochar was

prepared by pyrolysing PKS in a furnace at 500°C for 45 min before being quenched and flushed with distilled water until pH 8, and oven-dried at 105°C overnight. For the oxidation process, PKS biochar was soaked with 30% H<sub>2</sub>O<sub>2</sub> at a ratio of 1g: 1 mL for 12 hrs in a water bath at 95°C. After that, the oxidised PKS biochar was washed with distilled water until pH 7 and oven-dried at 105°C overnight [18].

## Physico-Chemical Characterisation

The proximate analysis for raw PKS, PKS biochar, PKS activated carbon, and oxidised PKS biochar was conducted by thermogravimetric analysis [21]. The fixed carbon content was calculated by the summation of percentages of moisture, ash, and volatile matter subtracted from 100 [22]. Surface functional groups were analysed using FTIR spectroscopy (Perkin Elmer, Spectrum One). The sample pellets were prepared by mixing and pressing about 1 mg of the sample with 100 mg potassium bromide. The FTIR spectrum was collected at the wavenumber range of 4000 cm<sup>-1</sup> to 450 cm<sup>-1</sup> with 64 scans and a resolution of 4 cm<sup>-1</sup>. For determining the surface area of raw PKS, PKS biochar, PKS activated carbon and oxidised PKS biochar, the iodine number [23] and BET (N<sub>2</sub>) methods [24] were adopted. Cation exchange capacity was also determined using the sodium acetate method [25]. The images of the microscopic structure of the surface of raw and oxidised PKS biochar were collected using a scanning electron microscopy (SEM) (Hitachi TM3030plus). Uncoated samples were mounted on the copper stub using double-stick carbon tape.

## RESULTS AND DISCUSSION

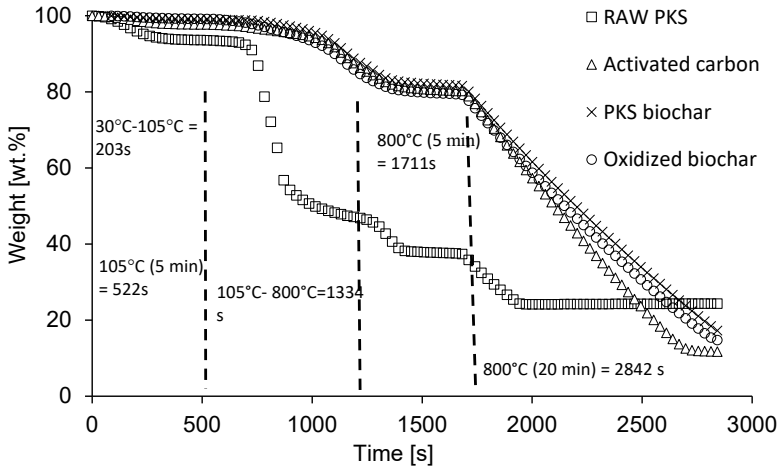
### Yields of PKS Biochar, PKS Activated Carbon and Oxidised PKS Biochar

The yield of PKS activated carbon was 44.5 ± 5 %, which was very similar to the values reported in the literature (43.4 %) [26]. The yields for PKS biochar and oxidised PKS biochar were 35 ± 7 % and 31 ± 1 %, respectively. The comparatively lower yields of the oxidised PKS biochar

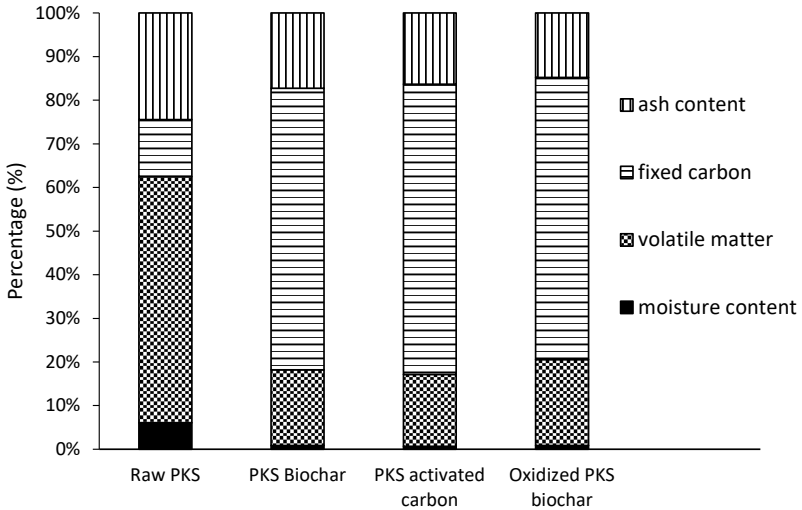
were expected since it has a lower ash content than that of PKS biochar. The ash may have been removed from the oxidised PKS biochar during the  $H_2O_2$  treatment process. Besides, chemical activation on PKS favours dehydration over depolymerisation reactions during pyrolysis that results in a larger yield for PKS activated carbon [27].

## Thermogravimetric Analysis

Figure 1 shows the thermogram for raw PKS, PKS biochar, PKS activated carbon and oxidised PKS biochar. During the first heating step (30-105°C), drying took place where moisture was removed. As shown in Figure 2, raw PKS has the highest moisture content (6.44 %) as it was not subjected to intense heat treatment. Pyrolysed samples (i.e. PKS biochar, PKS activated carbon and oxidised PKS biochar) contain minute quantities of moisture (all <1 %). The second heating step occurred between 105-800°C, where biomass constituents decompose into the volatile matter and form new aromatic carbon structures [22]. Raw PKS had the highest volatile matter content (60.1 % dry basis d.b.), similar to values reported in the literature (69.9 % d.b.) [28]. Despite being pyrolysed at 500°C, PKS biochar still contained 17.5 % (d.b.) volatile matter. In contrast, a volatile matter for the oxidised PKS biochar was 19.9 % (d.b.) suggesting that  $H_2O_2$  treatment did not remove substances that inhibited the volatilisation process. The PKS activated carbon was found to have a volatile matter content of 18.9 %. During the third heating step, fixed carbon was produced at 800°C. The final step was the combustion in an oxygenated environment at 850°C, whereby all fixed carbon content was oxidised, leaving only ash at the end of the process. As expected, pyrolysed samples (i.e. PKS biochar, PKS activated carbon and oxidised PKS biochar) had a high fixed carbon content (i.e. 65.1 - 68.5 %) compared to raw PKS (13.9 %). Carbonisation process removed oxygen functional groups via decarboxylation and decarbonylation [28] and increased carbon content in the pyrolysed samples. Figure 2 shows that raw PKS has a high ash content (26.0 %) while oxidised biochar had the lowest ash content (14.9 %), followed by PKS activated carbon (12.6 %) and PKS biochar (17.4 %). This was possible because the ash was leached from the oxidised PKS biochar, as it was twice rinsed with distilled water during its preparation.



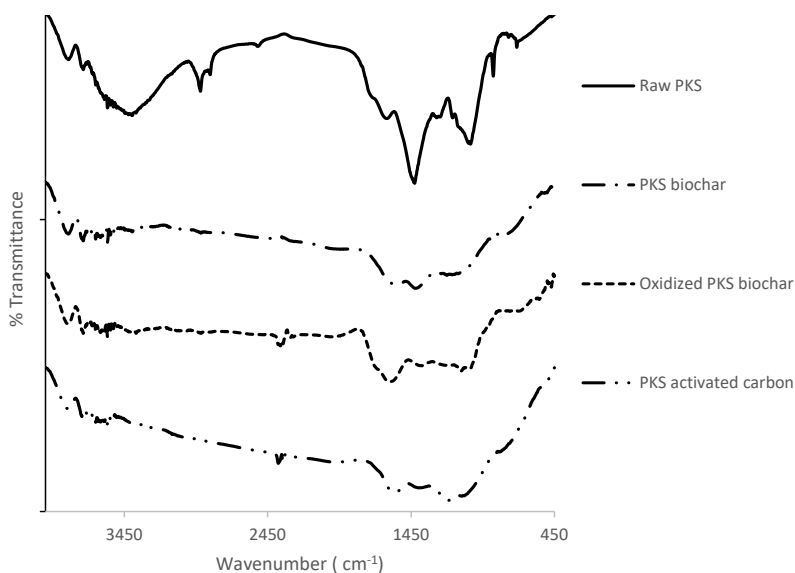
**Figure 1: Thermogram for PKS (Square), PKS Activated Carbon (Triangle), PKS Biochar (Cross), and Oxidised PKS Biochar (Circle)**



**Figure 2: Percentage (% w/w) of Moisture Content, Volatile Matter, Fixed Carbon, and Ash Content for Raw PKS, PKS Biochar, PKS Activated Carbon, and Oxidised PKS Biochar**

## Fourier Transform Infrared Spectroscopy

Figure 3 and Table 1 summarise all the functional groups present in raw PKS, PKS biochar, activated carbon and oxidised biochar. A broad and strong band at around  $3300\text{--}3400\text{ cm}^{-1}$  was observed in the FTIR spectrum for raw PKS. However, this band almost disappeared for PKS biochar, PKS activated carbon and oxidised PKS biochar. This band is attributed to the O-H stretching vibration for hydroxyl groups (i.e. phenolic, aliphatic hydroxyl) and carboxylic group [28]. A strong C-O stretch band at around  $1000\text{--}1300\text{ cm}^{-1}$  was observed in raw PKS spectrum, which indicates a high content of oxygen functional groups present in cellulose, lignin and hemicellulose [28]. This band is weaker in the spectra for pyrolysed samples (i.e. PKS biochar, PKS activated carbon, and oxidised PKS biochar) suggesting that decomposition of biomass constituents occurred. There is a strong and broad band of C=C stretching at  $1510\text{--}1650\text{ cm}^{-1}$  in the spectra of all pyrolysed PKS samples proving the presence of aromatic ring group [29]. Moreover, the band around  $1570\text{--}1515\text{ cm}^{-1}$  was due to the N-H bending of the amide group.



**Figure 3: FTIR Spectra of Raw PKS, PKS Biochar, Oxidised PKS Biochar and PKS Activated Carbon**

**Table 1: FTIR Bands in Raw PKS, PKS Biochar, PKS Activated Carbon and Oxidised PKS Biochar**

Vibration bands	Wavenumber (cm <sup>-1</sup> )			
	Raw PKS	PKS biochar	PKS activated carbon	Oxidised PKS biochar
O-H stretching	3394	-	-	-
C-O-H bending	1424	1421	-	-
C=C stretching	-	1537	1566	1597
C-O-C stretching	1031	1197	1159	1100, 1028
N-H bending	-	1537	1566	-

### Cation Exchange Capacity

The CEC measures the surface charge in a solid sample and indicates its capacity to adsorb cations [30]. Table 2 shows that raw PKS had the highest CEC value, followed by PKS activated carbon and oxidised PKS biochar. The lowest CEC value was recorded for PKS biochar. Raw PKS had several oxygen functional groups (i.e. hydroxyl, carboxyl) that contribute to a high CEC value. However, the PKS biochar has lost oxygen functional groups through decarboxylation and decarbonylation reactions that contributed to its lower CEC value. The H<sub>2</sub>O<sub>2</sub> oxidation process reversed this trend and doubled the CEC value of the oxidised PKS biochar (4.1 cmol/kg), due to the formation of new oxygen functional groups on its surface [31]. The CEC for PKS activated carbon (9.4 cmol/kg) is twice as much as that of the oxidised PKS biochar. The activation process may have introduced phosphate ion (PO<sub>4</sub><sup>3-</sup>) and increased the negative charge on the surface of PKS activated carbon. It should be noted that [32] reported that the content of alkaline ashes in biochars poses challenges to the accurate determination of CEC. The authors propose to pretreat biochars with diluted hydrochloric acid until the pH is near-neutral so that NH<sub>4</sub>OAc can buffer the biochar suspension effectively for subsequent CEC determination. Future research should thus be conducted to determine the CEC of biochars used in this study to establish whether alkaline elements affected the measurements.



**Table 2: Cation Exchange Capacity (CEC), Iodine Number and BET (N<sub>2</sub>) Surface Area for Raw PKS, PKS Activated Carbon, PKS Biochar and Oxidised PKS**

Biochar			
Type of sample	CEC (cmol/kg)	Iodine number (mg/g)	BET (N <sub>2</sub> ) (m <sup>2</sup> /g)
Raw PKS	10.8	61.5	N/D
PKS activated carbon	9.45	66.2	N/D
PKS biochar	2.33	65.6	1.44
Oxidised PKS biochar	4.10	65.7	0.12

### Iodine Number and BET (N<sub>2</sub>) Surface Area

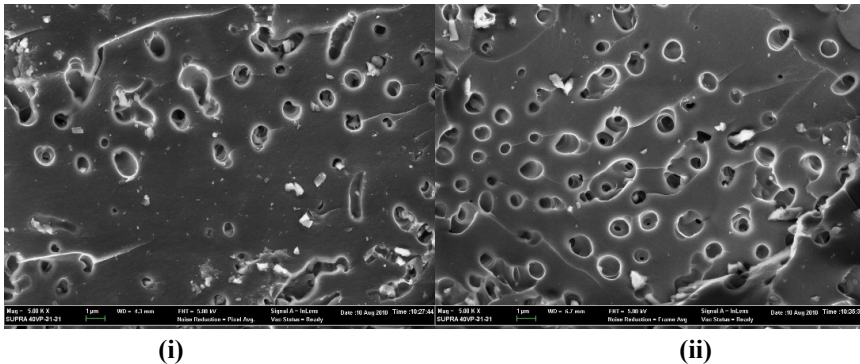
All three pyrolysed PKS samples had higher iodine numbers than raw PKS (Table 2). Activated carbon usually possesses high surface area due to higher contents of micropores for sorption of more iodine on its surface [33]. Treatment with H<sub>3</sub>PO<sub>4</sub> dehydrates PKS and develops micropores in the PKS activated carbon.

The specific surface area for PKS biochar (Table 2) was lower than the values reported in the literature. For example, [34] pyrolysed PKS at 500°C for 1 hr and obtained a BET (N<sub>2</sub>) surface area of 84 m<sup>2</sup>/g while [35] reported a value of 191 m<sup>2</sup>/g under similar pyrolysis temperature and holding time. [36] gasified PKS at 725 ± 75°C and reported a BET (N<sub>2</sub>) surface area of 23.7 m<sup>2</sup>/g. It is noteworthy that the volatile matter for PKS biochar (17.5 %) is also higher than that (12.3 %) reported by Lee *et al.* (2013) [35]. The presence of excess volatile matter in the PKS biochar may indicate the presence of tar that blocks the micropores causing the low specific surface area. The specific surface area for oxidised PKS biochar (0.12 m<sup>2</sup>/g) was even lower than that of PKS biochar.

The low surface area observed in this studies also suggests that the oxidation process may have only occurred on the outer region of the PKS biochar particles due to the presence of volatile matter in the form of secondary condensates which prevented H<sub>2</sub>O<sub>2</sub> from diffusing into the inner spheres of PKS biochar particles. This may explain why the iodine number has increased only marginally 37.

## Scanning electron micrography

Figure 4 shows SEM images for PKS biochar and oxidised PKS biochar. Compared to PKS biochar (i), the size of the outer pores of the oxidised PKS biochar (ii) was enlarged. The presence of enlarged pores showed that  $H_2O_2$  might have removed the pore-blocking compounds on the surface of PKS biochar. However, at 30% concentration,  $H_2O_2$  may have rapidly released an excessive amount of oxygen that destroyed the existing microstructure on the surface of PKS biochar. This is consistent with the finding in the specific surface area analysis, whereby the higher amount of macropore was the result of the oxidation reaction on the outer sphere of PKS.



**Figure 4: Scanning Electron Micrography Images for (i) PKS Biochar, and (ii) Oxidised PKS Biochar**

## CONCLUSION

Oxidation process has successfully increased the amount of polar oxygen functional groups on the surface of PKS biochar. However, the surface area of the oxidised PKS biochar is similar to that of PKS biochar and is still lower compared to that of PKS activated carbon. Based on the CEC and iodine number values, PKS activated carbon is still a better sorbent than oxidised PKS biochar. The fact that oxidised PKS biochar still contains

volatile matter proves that using 30 % H<sub>2</sub>O<sub>2</sub> was not enough to remove volatile organic compounds and improve the porous structure in the PKS biochar. Extreme thermal treatment is still required to remove the volatile matter from PKS to allow oxidation reaction to occur at the inner sphere of the PKS biochar particles. However, this method of producing sorbent from PKS remains economically and environmentally unsustainable due to the high energy input. Milder oxidants such as magnesium peroxide or calcium peroxide may be used to allow greater diffusion of oxidant into the PKS biochar particles and with a slower release of oxygen. This may enable uniform oxidation of PKS biochar, and possibly developing a greater amount of micropore and increase specific surface area.

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