The Characteristics of Oil Palm Shell Biochar and Activated Carbon Produced via Microwave Heating

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Abstract. Biochar was produced from oil palm shell via microwave-induced pyrolysis. The biochar was subsequently activated via microwave assisted CO_2 activation. A simple single layer arrangement of the microwave absorber (coconut shell based activated carbon) and oil palm shell in the reactor was adopted during pyrolysis. In recent times, the treatment of oil palm biomass using microwave heating technology has been on the increase. Value added products such as bio-oil, gas, biochar and activated carbon are being produced while at the same time serving as waste management control. Biochar is seen as a promising climate mitigation tool. Activated carbons can be used as absorbent for the removal of pollutants from wastewaters, as air pollution control and as electrode for supercapacitor. This paper presents comparative study between the characteristics of oil palm shell biochar and oil palm shell activated carbon. BET surface area and Scanning Electron Microscopy (SEM) were analyzed to establish the characteristics of the biochar and activated carbon.

Introduction

Malaysia being the second largest oil palm producer in the world with about 5 million hectare of oil palm plantation generates huge amount of oil palm biomass. The biomass, mainly palm oil mill effluent (POME), oil palm shell (OPS), mesocarp fibers and empty fruit bunches (EFB), is being generated as by-products from the 426 oil palm mills in Malaysia [1]. Traditionally, oil palm shell is used as feedstock for the boiler to generate electricity to power the mill. However, the mill still have excess amount of oil palm shell leftover from which value added products can be obtained in a sustainable approach.

Biochar is black solid residues obtain from the pyrolysis of biomass at moderate temperature in an inert environment (in the absence of oxygen). Pyrolysis is a thermal decomposition process. It is one of the recent renewable energy processes which if carefully controlled promises high yields of condensable liquids with minimum of gas and biochar. Biochar when use as a soil amendment play important role as a climate mitigation tool by capturing and storing carbon in soils.

Activated carbon which is mostly amorphous in nature can be produce from virtually all carbon based materials. However, agricultural waste is widely used as precursor material due to its abundant availability and relatively low cost. Activated carbon, due to its high surface area, tunable pore structure and low acid or base reactivity [2], find applications in a wide range of areas such as adsorbent for the removal of pollutants from wastewaters, air pollution control and electrode for supercapacitor. Preparation of activated carbons from carbonaceous materials is usually carried out either by physical activation process or chemical activation process. In chemical activation process precursor material is impregnated with chemical activation agent such as KOH, $ZnCl_2$, NaOH or H_3PO_4 and carbonization and activation is done in a single step. Chemical activation has the advantage of being performed at a lower activation temperature and in a shorter treatment time. In

physical activation process, environmental friendly activation agent such as steam, air, CO_2 or a combination of these gases is usually utilized. Physical activation has the problem of longer activation time and higher activation temperature resulting in higher energy cost and relatively lower yield. In the last two decades microwave (MW) heating technology has attracted attention of many researchers for the preparation of activated carbon because it offers a number of advantages such as shorter activation time and hence low energy cost, high heating rate, selective heating, uniform interior heating, reduced equipment size and greater control of heating process. The properties and quality of activated carbon greatly depend on the properties of the precursor material, the activating agent and the preparation conditions.

Several methods have been adopted for the preparation of activated carbon from oil palm shell. However, no work has been reported on microwave-induced CO_2 activation of oil palm shell. Also, there is no reported work on the use of biochar obtained from microwave pyrolysis of oil palm shell as precursor for preparation of activated carbon. This paper attempts to compare the characteristics of the biochar obtained from microwave pyrolysis of oil palm shell and the activated carbon produced by microwave-induced CO_2 activation of the biochar. Biochar and activated carbon were characterized using yield, scanning electron microscope (SEM) and surface area (BET).

Experimental

Materials. Oil palm shell (OPS) biomass received from a palm oil mill situated in Johor State, Malaysia was sieved to obtain particle size ranging from 2 mm – 4.75 mm. Laju Group of Companies, Malaysia supplied the commercial coconut-based activated carbon (CAC) used as microwave absorber. The sized of CAC ranges from 1 mm – 1.8 mm. The proximate analysis of OPS at dry condition reported by [3] is volatiles 78 wt.%, fixed carbon 20 wt.% and ash content 2 wt.%; as received OPS has inherent moisture content of about 8 wt.%. The microwave system consists of modified multimode domestic microwave (1 kW, 2500 MHz), fluidized bed glass quartz reactor (5 cm internal dia. X 20 cm height) with wire mesh which serves as screen support as well as distributor plate, N_2 and CO_2 gases, K-type thermocouple (0 – 1200 ^oC range) inserted into the bed region and data acquisition system linked to personal computer for continuous monitoring and recording of process temperature.

Preparation of biochar and activated carbon. For the pyrolysis of OPS, a single layer arrangement of precursor and CAC in the reactor was adopted. OPS/CAC ratio and irradiation time were fixed at 1:0.5 and 25 min respectively while microwave power of 300 W, 450 W and 600 W were selected for the pyrolysis. CAC was uniformly distributed on the wire mesh followed by OPS. N_2 was supplied at a flow rate of 10 LPM for about 5 min before commencement of experiments to ensure inert environment and was reduced to 5 LPM during experiment so as to maintain the inert environment and also to sweep out the vapor. Activation of the resultant biochar was performed under a CO_2 of flow rate 2 LPM at microwave power of 450 W and activation time of 15 min. Activated carbon samples were allowed to cool down under inert environment to room temperature.

Characterization. Scanning electron microscopy (SEM) analysis with Philip XL40 was carried out for biochar and activated carbon to study the development of surface morphology and porosity. Surface physical properties were characterized with gas adsorption porosimeter (Thermo Scientific Surfer, Italy). Brunauer-Emmett-Teller (BET) equation was used to calculate the BET surface area.

Results and Discussion

Temperature profiles. Fig. 1 shows real time bed temperature profiles recorded during pyrolysis of the oil palm shell. It can be observed that the temperature profiles are similar to each other with almost the same pattern; MW power of 300 W has a more pronounce temperature profile. For all MW powers, the maximum temperature was achieved before 6 min after which the temperature started dropping suggests that the volatiles components in the OPS have been vaporized between 0

and 6 min and the pyrolysis process completed, hence the continuous drop in temperature till the end of experiment. The cyclic ON/OFF of the magnetron in the multimode domestic microwave is responsible for the sinusoidal nature of the temperature profiles observed in Fig. 1. This variable output in multimode domestic microwave is capable of distorting the exact temperature of the material.

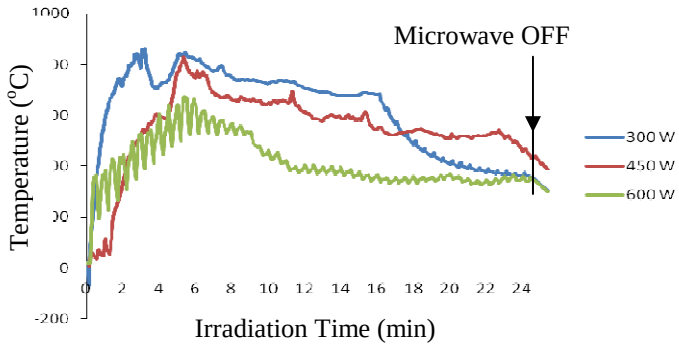


Fig. 1 Real time bed temperature profiles of OPS biochar

During activation process, monitoring of process temperature was difficult due to temperature ramp beyond the range of the thermocouple leading to the damage of thermocouple.

Effect of pyrolysis conditions on biochar and activated carbon yield. The biochar and activated carbon yield at different microwave power is shown in Fig. 2. Activated carbon yield obtained for all power level ranges from 92.5 wt% to 93.3 wt%. Longer residence time during pyrolysis contributed immensely to the volatilization of cellulose, hemicelluloses and lignin (volatiles) which may be responsible for the low char yield. The effect of radiation time has overshadowed the effect of microwave power hence only slight difference was observed in the percentage yield.

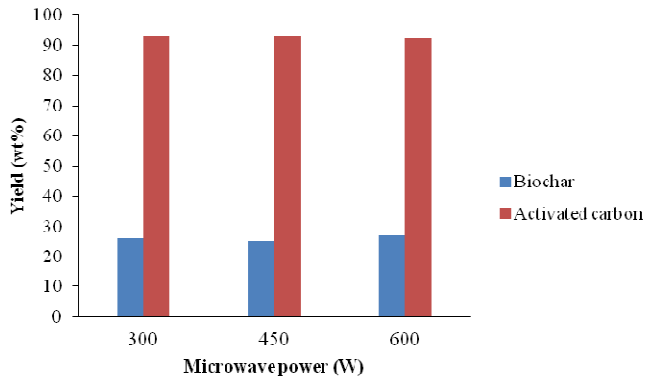
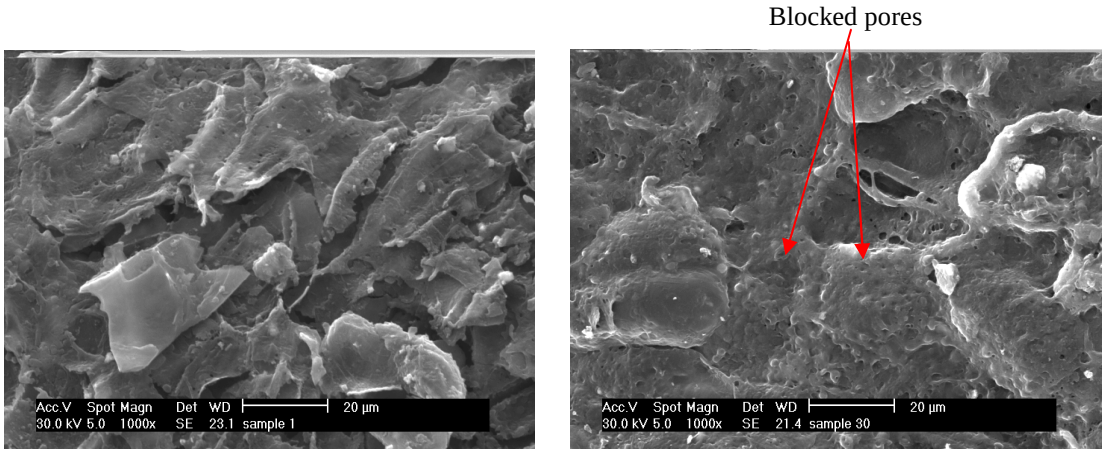


Fig. 2 Biochar and activated carbon yield from OPS biomass and biochar respectively.

Scanning Eletron Micrograph. The SEM images of biochar and the resulting activated carbon are presented in Fig. 3. The surface morphology varies significantly with biochar showing cracks and no visible pore development, while the image of the activated carbon shows the formation of some cavities and rudimentary pores as a result of the space created by the volatilization of the moisture, hemicelluloses, cellulose and lignin content and the effect of the reaction of the CO_2 and the walls of the carbon. However, most of the pores are constricted and blocked; this may be as a result of the high temperature and sinusoidal heating effect of the magnetron. Nevertheless, efforts are been made to abate this phenomenon in our future work.

Surface Area and Pore Volume. The surface area is an important property of activated carbon which to a great extent determines its adsorption capacity and type of application. BET surface area and pore volume of oil palm shell derived biochar and activated carbon are presented in Table 1.



(a) (b)

Fig. 3 SEM images of (a) biochar and (b) activated carbon

The results are comparable with those reported by previous researchers e.g. [4]. The pore structure of the activated carbon is predominantly micropores with about 86% of total pore volume.

Table 1: Surface area and pore size characterization of biochar and activated carbon

Properties	Biochar	Activated Carbon
BET surface area (m^2/g)	23	151
Micropore volume (cm^3/g)	0.0093	0.077
Mesopore volume (cm^3/g)	0.0057	0.012
Total pore volume (cm^3/g)	0.015	0.089

Conclusion

Pyrolysis conditions of the oil palm shell under microwave heating have been found to be important factors that influence the physical characteristics of the biochar and the resulting activated carbon. The present study has shown that longer pyrolysis time is tantamount to energy wasting and detrimental to the development of pore structure. Reduction in radiation time is expected to bring about considerable reduction in cost of production as well as improvement in the pore structure.

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