

Preparation of Activated Carbon from *Parkia Speciosa* Pod by Chemical Activation

P.Y.L Foo, L.Y. Lee

Abstract— *Parkia speciosa* pod which is known as stink bean or 'petai', is selected as a precursor for activated carbon preparation by activation with phosphoric acid. The effects of carbonisation temperatures ranging from 450°C to 650°C and mass ratio of acid to precursor (1:1 and 1:2) on the porosity formation of the resulting carbons are studied for a carbonization period of 1 hour, heating rate of 5°C/min and nitrogen purge rate of 10 cm³/min. Carbon yield is determined by measuring the weight of a sample before and after carbonisation. The surface area and pore volume are measured using the Micromeritics 2020 surface analyser. The maximum surface area of 190 m²/g and pore volume of 0.0950 cm³/g are obtained at carbonization temperature of 600°C and impregnation ratio of 1:1. The measured carbon yield, which was comparable with that of a commercial activated carbon, was 41.1 %.

Index Terms— Activated carbon, agricultural, chemical activation, phosphoric acid.

I. INTRODUCTION

Activated carbon is one of the most popular adsorbents used in numerous industries for the removal and recovery of organic and inorganic compounds from gaseous and liquid streams. It has high adsorption capability due to its high internal surface area and porosity formed during carbonization process. The presence of activating agents and carbonisation conditions influenced the development of pore structures [1]. However, the usage of activated carbon is limited by its high commercial cost which is a consequence of high production cost. Agricultural wastes are potential precursors because of the abundant supply and low cost. Moreover, transforming agricultural wastes into valuable end products may help to increase crop yields and production efficiency as well as to reduce solid wastes disposal problem. Many attempts have been made to produce activated carbon with agricultural wastes such as macadamia nut-shell [2], apricot stones [3], coconut shells [4], sugarcane bagasse [5], Ceiba pentandra hulls [6], corn cob [7] and sago waste [8]. But, to-date, no work has been

published on *Parkia speciosa* known as petai in Malaysia. *Parkia speciosa* seeds are used as a main ingredient in cooking and have always been a popular local delicacy whereas the pods are usually disposed. Hence, *Parkia speciosa* pods can be a potential precursor to produce activated carbon.

The methods used for activated carbon synthesis are physical activation and chemical activation. In physical activation, the precursor is first carbonised in an inert atmosphere and then activated in a stream of carbon dioxide or steam; whereas for chemical activation, the precursor is impregnated with a dehydrating agent, usually zinc chloride or inorganic acids, prior to carbonization in an inert environment. In this study, chemical activation method is used due to the fact that the chemical activating agents play an important role in the carbonisation process, in which they act as dehydration agents and minimize the formation of tar during carbonisation. Furthermore, the temperature range used in chemical activation is lower in comparison to that used in physical activation [9]. Phosphoric acid is selected as the activating agent instead of zinc chloride so as not to aggravate environmental pollution by contamination with zinc compounds and also it is easier to recover the carbon product during processing stage i.e. only rinsing with water is required [10]

The main objective of this research is to prepare activated carbon from the pods of *Parkia Speciosa*. A suitable preparation conditions including carbonisation temperatures and acid impregnation ratio is to be established.

II. EXPERIMENTAL

A. Pretreatment

Parkia speciosa was obtained from a local market and the seeds were taken out from the pods. The pods were repeatedly washed with distilled water to remove residues and dried at 100°C for 24 hours to reduce moisture content. The dried peels were ground with a commercial miller and then sieved (Endecotts Ltd.) to a particle size of 125 micrometer in diameter.

B. Impregnation

To obtain an impregnation mass ratio of 1:1, 10g of *Parkia speciosa* pods particles was poured into a beaker containing 7.9 ml phosphoric acid of 85wt% purity. The volume of the mixture was topped up to 35ml by addition of distilled water. Then the mixture was stirred thoroughly before being dried in the oven (Mettler) at 110°C for 24 hours. The procedure was repeated whereby 10g of precursor was mixed with the appropriate volume of acid to

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give an impregnation ratio of 2:1.

C. Carbonization

After drying, the samples were placed in crucibles and into a horizontal tubular furnace (Carbolite) for carbonization. The carbonisation process was carried out in an inert atmosphere using nitrogen gas of 99.9% purity, supplied at a rate of 100 ml/min STP. After adjusting the nitrogen flow, the tubular furnace was programmed with the ramp rate of 5oC/min. Once the desired carbonization temperature was attained, the heated samples were held isothermally at the set point for 1 h before cooling down to room temperature under nitrogen flow for 24 hours. The carbonization temperatures used were 450, 500, 550, 600 and 650°C. For the washing process, 1M KOH was used for cleansing purpose. 50 ml of KOH was poured into a beaker containing the cooled carbonised sample. The mixture was stirred thoroughly to remove the excess chemicals and then solids were separated using filter paper. Subsequently, the samples were rinsed sequentially with warm distilled water, followed by cold distilled water until the pH of the wash solution fell in the range of 6-6.5. The solution pH was measured using a bench-top pH meter (MERCK). The resultant samples were dried at 105°C in the oven (Memmert) for 24 hours.

D. Characterization of activated carbon

The surface area and micropore analysis were obtained from nitrogen adsorption at -196°C using a porosimeter (Micromeritics 2010). BET surface areas of the activated carbon samples were then determined using the Brunauer-Emmett-Teller equation assuming that the surface area occupied by per physisorped nitrogen molecule was 0.162nm². The nitrogen adsorption data was recorded automatically and presented as a plot of volume of nitrogen adsorbed versus relative pressure.

Approximately 0.1g of each corresponding activated sample was measured and degassed under vacuum (300mmHg) at a temperature of 300°C for 10 hours to remove excessive moisture and contaminants. The degassed sample was then cooled and weighted. The surface area analysis was performed automatically once the weighted samples had been transferred to the analysis port. The data were obtained through the control module and the review of the status of the system can be performed on the control screen.

III. RESULTS AND DISCUSSION

A. Effect of carbonization temperature on surface area and pore volume

Fig. 1 shows the BET surface area of the prepared activated carbon (AC), calculated using nitrogen isotherms at -196°C, at different carbonization temperatures using impregnation ratio of 1:1 and 2:1. The surface area increases gradually from 450°C to 600°C and it starts to decrease from 650°C onwards. At low temperatures, activation is not completed due to slow reaction rate between the chemical activating agent and carbonized material and hence, activation temperature below 450°C looks impractical. As temperature increases, the porosity and surface area increases due to the release of tars from cross-linked

framework generated by the treatment of phosphoric acid [11]. The highest surface area was 189.0 m²/g developed at an activation temperature of 600°C. At temperatures beyond 650°C, surface area decreases which may be due to the collapse of micropores formed during previous stages. It can be observed that at all carbonization temperature from 450°C to 650°C, impregnation ratio of 1:1 gives a higher surface area compared to impregnation ratio of 2:1.

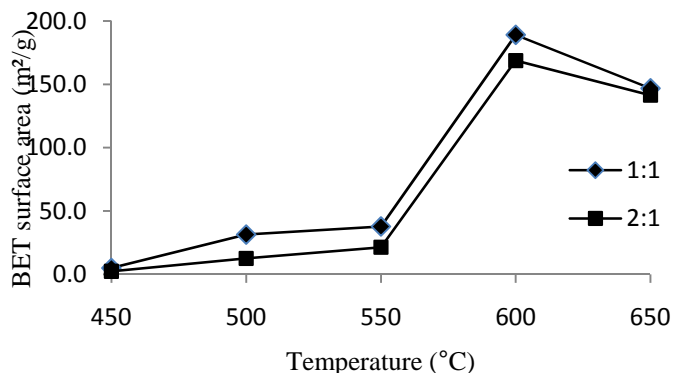


Fig. 1. BET surface area in relation to carbonization temperature.

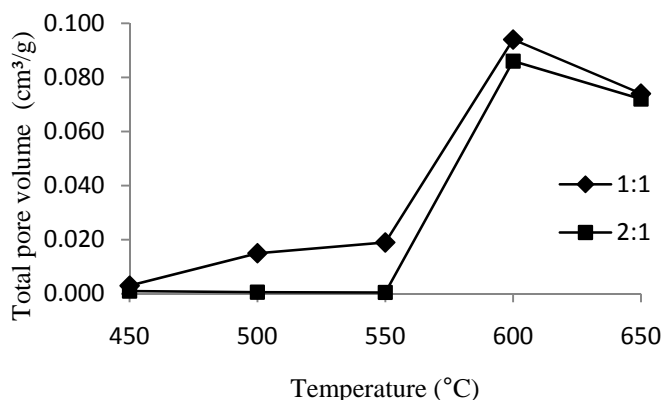


Fig. 2. Total pore volume in relation to carbonization temperature.

Fig. 2 shows the effect of temperature on the development of total pore volume of AC. The results indicated that an increase in temperature resulted in an increase in total pore volume. The carbon with highest pore volume was 0.094 cm³/g at 600 °C for impregnation ratio 1:1 and 0.086 cm³/g at 600 °C for impregnation ratio 2:1. Further increase in temperature at 650°C resulted in a decrease in total pore volume to 0.074 cm³/g and 0.072 cm³/g respectively. Beyond a certain carbonization temperature, the surface area and pore volume decreases due to excessive carbon burn-off. This leads to the widening of pores and destruction of some walls between the pores and hence, there is a reduction in porosity [12]. Similar results have been reported by other researchers [13-15].

B. Effect of carbonization temperature on activated carbon yield

Carbonization temperature plays a significant role on the activated carbon yield. The variation of AC yield with carbonization temperature is shown in Fig. 3. Generally, impregnation ratio of 1:1 gives a higher yield compared to impregnation ratio of 2:1. For both impregnation ratio, it was found that the yield of activated carbon decreases with

increasing temperature. This was due to the loss of most of the volatile matter and loss of phosphate compounds [16]. Under high activation temperature, the porosity and surface area decreased tremendously as a considerable amount of phosphate compounds volatilised when the carbonization temperature is above 650°C. Phosphoric acid acts in two different ways during the chemical activation of lignocellulosic feedstock. It promotes the rupture of bonds in the biopolymer constituents and forms cross-links through cyclization and condensation processes [9]. In addition, phosphoric acid combines with organic species forming phosphate and polyphosphate bridges that connect biopolymer fragments, thereby partially hindering the contraction in the material as the temperature increases [17]. Above 650°C, the phosphate and polyphosphate bridges become thermally unstable, their loss producing an increase in the size of aromatic units and a contraction in the material, which will result in a decrease of porosity.

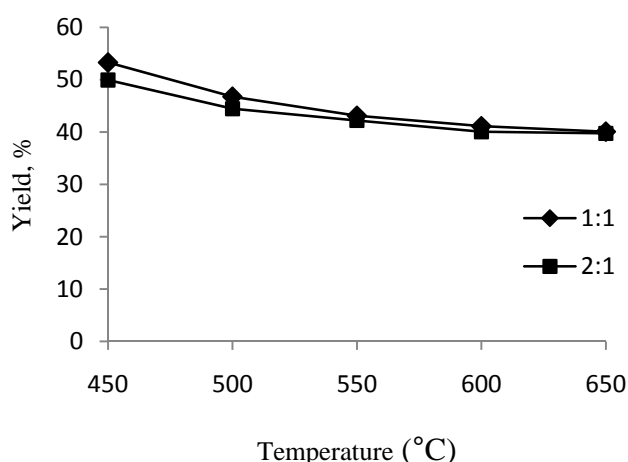


Fig. 3. Activated carbon yield in relation to carbonization temperature.

IV. CONCLUSION

Activated carbons with relatively high surface areas were prepared from *Parkia speciosa* pods with chemical activation. The surface area and pore volume of the activated carbon are heavily dependent on the carbonization temperature at which the carbon was prepared. Impregnation ratio of 1:1 produced activated carbon with higher BET surface area compared to activated carbon prepared using impregnation ratio of 2:1. Higher carbonization temperature would produce activated carbon with higher surface area and pore volume. However, yield of AC decreases as carbonization temperature increases.

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