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FABRICATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM SWAMP TARO STALK USING H_3PO_4 ACTIVATION

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ABSTRACT

In this study, highly mesoporous activated carbon was prepared from swamp taro stalk via single step H_3PO_4 activation. The effect of impregnation time (1 – 3 h) on the yield and surface area of activated carbon was investigated. The synthesized activated carbon was examined by Fourier Transform Infrared Spectroscopy (FTIR) and N_2 adsorption-desorption analyses. The optimization investigation clearly showed that the impregnation time gives significant effect on the yield and BET surface area of the materials. Under the optimum impregnation time of 2 h, the registered yield percentage and BET surface area were 35.82 and 1390.41 m^2g^{-1} respectively. Type IV isotherm with H1 loops obtained from N_2-sorption studies indicates the fairly narrow mesoporous network structure. This study proved the facile synthesis route of high grade activated carbon from waste biomass.

Keywords: Activated carbon; H_3PO_4 activation; Stalk; Single step; Swamp taro

INTRODUCTION

Activated carbon is a carbonaceous material which exhibits large specific surface area and high pore volume. They are powerful adsorbent which being widely applied in wastewater treatment. However, industrial manufactures struggle with the high production cost and less avaibility of raw materials. Hence, it creates awareness for the researchers to seek for readily available and low cost raw materials for the production of activated carbon. Previous studies reported that plant waste containing lignocelluloses from agricultural and forestry is reproducible and cheap resources, which can be used as raw materials to synthesize good grade activated carbon. Thus, swamp taro stalk was found to be a cheap and highly available precursor for the fabrication of activated carbon [1]. Furthermore, this plant possesses no commercial value and this stipulated us to utilize it for the production of high quality activated carbon.

Fabrication of activated carbon can be done either via physical or chemical activation. In comparison to physical activation, chemical activation method exhibit advantages such as save time, reduce cost, higher product yield and low energy consumption [2]. This method involves thermal decomposition of the char impregnated with dehydrating agent such as H_3PO_4, NaOH, KOH, $NaCO_3$ and $ZnCl_2$ followed by heat treatment at moderate temperature ranging from 400 ºC to 700 ºC [3]. Amongst numerous chemical activating agents, H_3PO_4 are widely used for lignocellulosic materials due to good dehydration effect as claimed by Martin-Gonzales et al., [4]. Furthermore, the chemical activator can produce activated carbon with higher yield by formation of polyphosphate layer which protect the internal pore structure from excessive burn-off [5].

Therefore, the objective of this work is to fabricate activated carbon from swamp taro stalk via single step H_3PO_4 activation. The effect of the impregnation time on the yield and BET surface area was investigated. The product obtained was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and N_2 adsorption-desorption analyzer.

EXPERIMENTAL

Materials

Swamp taro stalks were collected from local swamp at Arau, Perlis, Malaysia and used as raw materials. Phosphoric acid (Acros, 99.0%) was used as the chemical activating agent. The chemicals received were used without any further purification.

Preparation of activated carbon

The stalk was cut, dried (60 ºC), grind and sieved to the size of 212 µm. The sample was impregnated with H_3PO_4 in a 1:1.5 (taro stalk/H_3PO_4 w/w) under stirring (230 rpm) at 75 ºC for 2 h. Then, the sample was dried at 80 ºC and heated in the muffle furnace at 500 ºC for 30 min with a heating rate of 3 ºCmin^{-1}. Later, the product was crushed, washed until pH 7, dried at 110 ºC for 4 h and sieved to the size of 150 µm. The product was weighed and denoted as H_3PO_4-AC. The yield percentage of the product was calculated based on following equation.

$$\text{Yield } \% = \frac{M_{Final}}{M_{Initial}} \text{ x } 100\%$$

$$M_{Final} = \text{Mass of product}$$

$$M_{Initial} = \text{Mass of precursor}$$

In this work, impregnation time (1 - 3 h) was optimized in order to achieve maximum BET surface area and yield %.

Sample characterization

The swamp taro stalk and synthesized activated carbon were characterized by Fourier Transform Infrared Spectroscopy (FTIR, Thermo Electron Corporation Nicholet 380). Synthesized activated carbon was further characterized by N_2 adsorption-desorption analyzer (Micromeritic ASAP 2020).

RESULTS AND DISCUSSION

Figure 1 shows the effect of impregnation time on the yield percentage and BET surface area of H_3PO_4-AC.

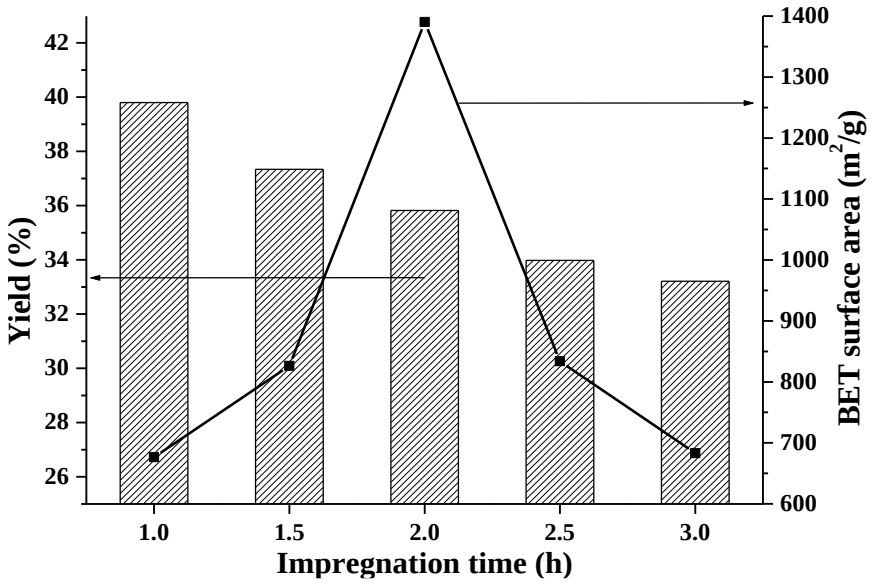

Figure 1: Effect of the impregnation time on the yield and BET surface area of activated carbon

Referring Figure 1, product yield decreased upon an increase in impregnation time due to the release of volatile matter and reaction with H_3PO_4. This phenomenon contributed to the formation of pore which correlates with the increasing trend in BET surface area. The trend of graph was similar with the work of Ozdemir et al., [6] over grape stalk. This occurrence presumably due to the reaction of precursor with H_3PO_4 begins as soon as the components are mixed at 75 ºC impregnation temperature. Thus, increasing the impregnation time might have provided longer contact between H_3PO_4 and the precursor to react. According to Marsh & Rodriguez., [7], first stage of reaction between H_3PO_4 and raw materials start at 50 ºC. At this stage, the acid starts to attack hemicellulose and lignin which possibly due to easier access to these amorphous polymers than crystalline cellulose. The primary effects of acid attacks are to hydrolyze glycosidic linkages in the hemicellulose and to cleave aryl-ether bonds in the lignin. These reactions are followed by dehydration, degradation and condensation. Hence, more volatile matter will be gasified and accompanied with the formation of pores.

Nonetheless, tremendous reduction in the BET surface area was observed at further impregnation (>2 h) which may due to the destruction of pores resulting from the excessive dehydration.

Fourier Transform Infrared Spectroscopy

Figure 2 compares the FTIR spectra of the raw swamp taro stalk and the prepared H_3PO_4-AC.

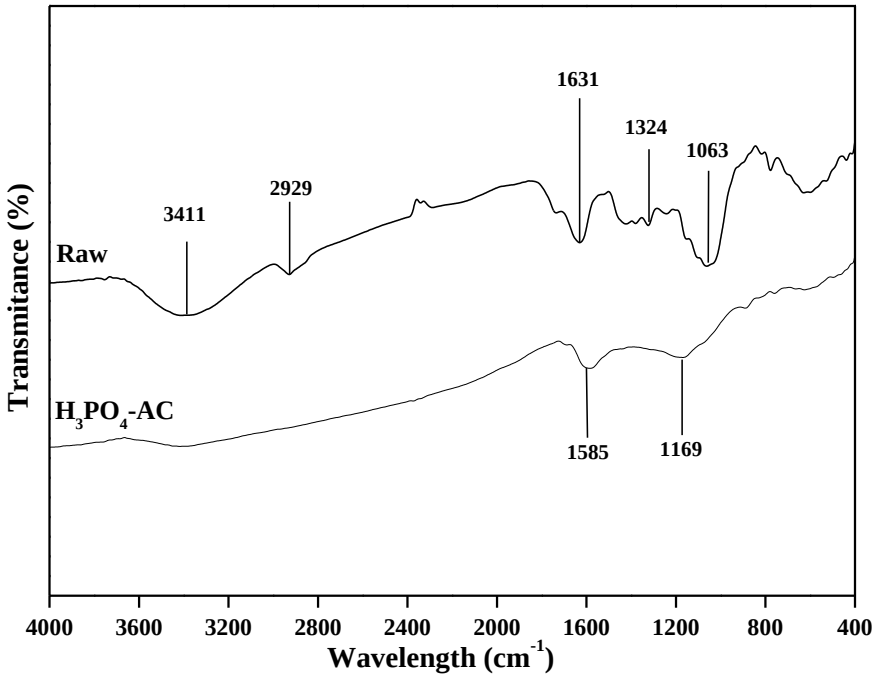

Figure 2: FTIR spectra of swamp taro stalk and H_3PO_4-AC

Based on the FTIR spectra in Figure 1, swamp taro stalk exhibits large number of adsorption peak which indicated the complex nature of the materials. This observation was expected as swamp taro stalk is lignocellulosic material which is made up with large numbers of functional groups. A broad peaks at 3411 cm^{-1} indicates the H-bonded of O-H stretching vibration of absorbed moisture, hemicellulose, cellulose and lignin [8]. Furthermore, a small peak at 2929 cm^{-1} and 1324 cm^{-1} attributed to the aliphatic saturated C-H stretching and bending vibration in lignin polysaccharide includes cellulose and hemicellulose [9]. However, these bands disappeared in the FTIR spectra of H_3PO_4-AC, Kilic et al., [10] claimed the phenomenon may due to the removal of volatile matter during chemical activation. The weak band in 1585-1631 cm^{-1} showed the presence of C=C stretching vibration of aromatic hydrocarbon in lignin [11]. A strong band in 1063-1169 cm^{-1} corresponded to O-C stretching vibration of alcohol, carboxylic acid, ether and ester [8]. In addition, for the FTIR spectra of H_3PO_4-AC, the band can be also assigned the O-C stretching vibration of P-O-C linkage and P=OOH bonded due to the formation of phosphate bridge during activation [7, 8]. The dissapearance of some functionals groups in H_3PO_4-AC indicates the dehydration of

volatile matters during the chemical and thermal treatment. Interestingly, the formation of phosphate group can also act as surface functional groups which reflect the good feature of the chemical activating agent.

N_2 adsorption-desorption isotherm

The N_2 adsorption-desorption isotherm and hysteresis loops of the synthesized material is illustrated in the Figure 3.

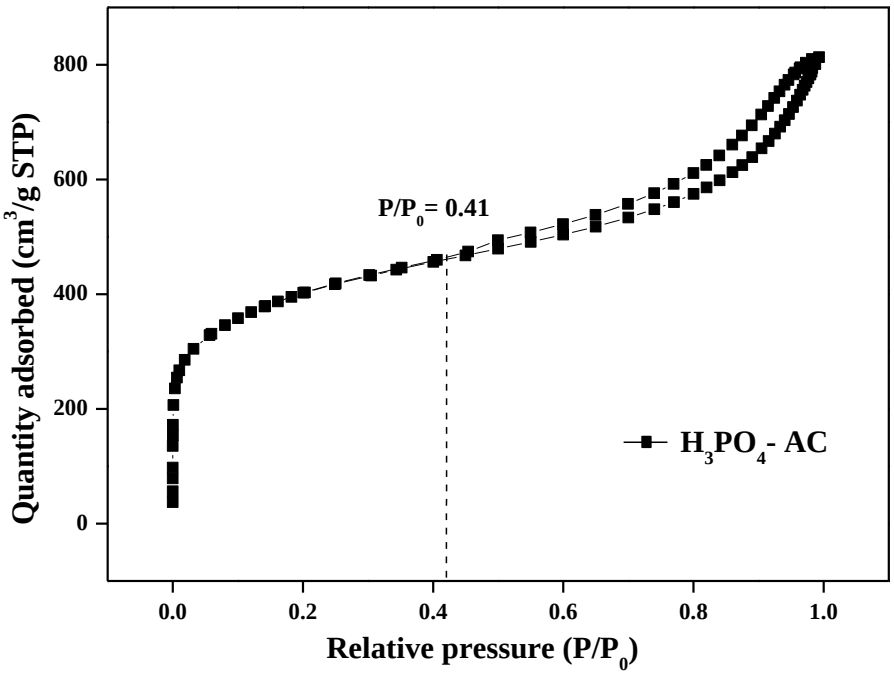

Figure 3: N_2 adsorption-desorption isotherm of H_3PO_4-AC

Referring Figure 3, H_3PO_4-AC shows Type IV isotherm which is a characteristics of mesoporous network structure [12]. Based on Blanco et al., [13], the loop of isotherm in the range of $0.28<P/P_0<0.43$ corresponded to consistency of mesopores size. Furthermore, H1 hysteresis loop was observed which represents the uniform and fairly narrow porous structure related to capillary condensation in open-ended cylindrical pore [14].

CONCLUSION

In this work, low cost biomass was successfully converted into a benign product. Impregnation time has affected the yield % and BET surface area of the prepared activated carbon. Under the optimum impregnation time (2 h), percent yield and BET surface area were 35.82 and 1390.41 m^2g^{-1} respectively.

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