

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON DERIVED FROM MANGOSTEEN PEEL IN TREATING BENZENE VAPOUR

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1.0 Introduction

Adsorbents are required in many different applications, in either liquid or gas phase processes. Activated carbons are widely used as adsorbents in technologies related to pollution abatement due to their highly porous texture and large adsorption capacity (Lozano-Castello *et al.*, 2001). The continuously increasing list of environmental concerns and the interest in utilization of various wastes have awakened the interest for carbon adsorbents based on agricultural and forest wastes. Agricultural by-product such as coconut shell (W.Li *et al.*, 2009), grain sorghum (Y.Diao *et al.*, 2002), coffee bean husk (L.C.A. Oliveira, *et al.*, 2009), rubber wood sawdust (C.Srinivasakannan *et al.*, 2004), guava seed (Elizalde-Gonzalez, 2009) and fruit stones have been discovered to be suitable precursors for activated carbon production. The choice of precursors largely dependent on its availability, cost and purity but the manufacturing and intended application of the product are also important considerations. Basically, there are two different processes for preparation of activated carbon, either physical activation or chemical activation. Physical activation implies two-step scheme by carbonization and subsequent partial gasification with CO₂ or air at temperature range of 800- 1000°C while chemical activation is carried out in only in one stage at lower temperature range of (450-700°C) using chemicals such as ZnCl₂, H₃PO₄ and KOH.

2.0 Objective of the research

1. To study the physical and chemical characteristics of mangosteen peel
2. To prepare high surface area activated carbon from mangosteen peel by optimizing the condition (impregnation ratio and activation time)

3.0 METHODOLOGY

3.1 Collection and size reduction of sample

Mangosteen peel was obtained from Jusco, Serdang. The starting material was initially washed thoroughly with distilled water to remove dirt and other impurities. Then the peel were cut to obtain particle size of 1-2 cm and dried at 70 ° C for 2 hours to reduce the moisture content.

3.2 Ultimate and proximate analysis

Ultimate analysis was carried out using Elemental Analyzer (932) and the oxygen calculated by differences. Proximate analysis consist of ash, moisture, volatile matter was carried out using AOAC method. Moisture content was determined by oven-drying the material at 110°C until consistency of weight was obtained. Ash content was calculated by heating the sample at 550°C in a muffle furnace for 2 hours until no mass variation.

3.3 TGA and FTIR

Mangosteen was subjected to thermogravimetric analysis (TGA) in a N₂ atmosphere using a thermal analyzer (TGA/STDA 851°). FTIR was performed by using spectroscopy apparatus to determine presence of bulk functional groups at room temperature.

3.4 Preparation of activated carbon by chemical activation

10g of dried mangosteen peel was soaked with 100ml of acid with different concentration. Then, dried impregnated material was dried in oven for 12 hours at 100 °C. After that, the dried material were activated by using nitrogen gas which was provided for the whole of activation process with heating rate 5°C/min and flow rate of 150cc/min. After activation, samples were cooled down to room temperature. The obtained activated carbon was thoroughly washed with distilled water until the wash liquor pH reading is at 6–7. On the average, 7–8 washings were able to neutralize the sample. The activated carbon will be washed with 0.1 M sodium hydroxide solution and finally with distilled water. Then the samples were dried in oven for 24 hours at 70°C and store in dessicator.

3.5 Characterization of activated carbon

The porous structure of produce activated carbon was determined by using Micromeritics ASAP 2010 instrument. The specific surface area was determined by using BET (Brunauer, Emmett and Teller) method.

3.6 Adsorption of pollutant

The activated carbons with high surface area were selected for the adsorption part. Sample consist of 1.0g activated carbon were enclosed in a gas sampling bag and residual air in the bag was degassed using a small air pump for few minutes. Benzene solvent was directly injected into tedlar bag consist of 5L of nitrogen which simulating likely service conditions in air purification processes for industrial occupations. The contact time started on injection of the benzene solvent.

4.0 Results and Discussion

4.1 Raw material characterization

4.1.1 Ultimate and proximate analysis

The results of proximate and ultimate analyse for mangosteen used in this study are given in Table 1 and 2. This result implies that mangosteen peel can be used as potential precursors for activated carbon production due to high carbon content and low ash production. The results are similar to activated carbon produced from jackfruit peel using H_3PO_4 (D. Prahast *et al.*, 2008)

Table 1: Ultimate analysis for mangosteen peel

Ultimate analysis (%)				
C	H	N	S	O
39.1	5.5	1.0	0.03	54.37

Table 2: Proximate analysis for mangosteen peel

Proximate analysis (%)			
Ash content	Moisture content	Volatile matter	Fixed carbon
9.9	22.8	12.9	54.4

4.2 Activated carbon characterization

4.2.1 Effect of acid concentration on yield of activated carbon

Figure 1 illustrates the effect of acid concentration on the yield of activated carbon produced. The yields of activated carbon produced were in the range of 30.78-33.88%. For the 10% acid concentrations, the yield observed was 33.88% and were decreased to 30.78% for 20% acid concentration. This is due to the reaction of phosphoric with the char and volatile matters are diffused quickly out of the surface particle during the activation process (P.Patnukao and P.Pavasant, 2008). Therefore, increment in acid content cause the gasification of surface carbon atoms become more predominant leading to an increase in the weight loss and yield of activated carbon. However, the yield was slightly increased when the concentration was up to 30-50%. The presence of higher acid content during activation promotes depolymerization, dehydration, and redistribution of constituent biopolymers, and also favoring the conversion of aliphatic to aromatic compounds thus increasing the yield of activated carbon (D.Prahast, 2008).

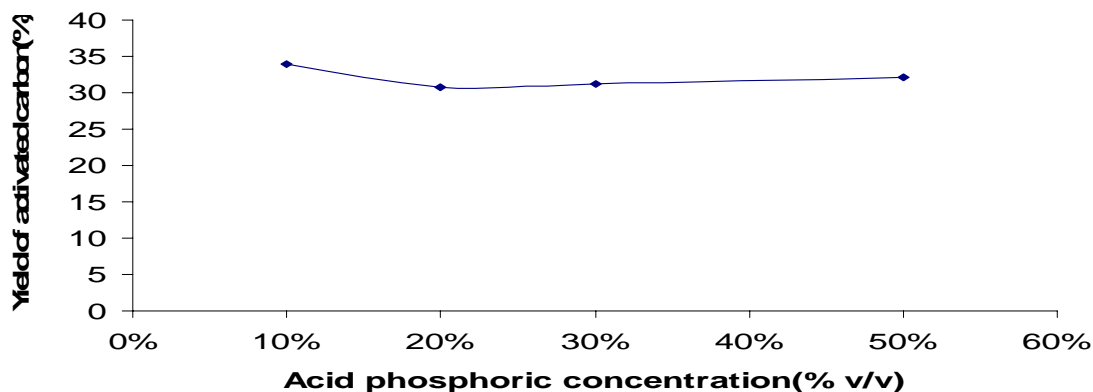


Figure 1: Yield of activated carbon at different acid concentration

Table 3: Pore size distribution of activated carbon derived from different acid concentration

Sample	S _{BET} (m ² /g)	Micropore surface area (m ² /g)	Micropore volume (cm ³ /g)	External surface area (m ² /g)	Average pore diameter(A)
Raw material	0.6598	-0.1931	-0.000113	0.9458	3.6757
Commercial AC	847.4407	499.8299	0.261373	347.6108	-
AC-10%	143.7346	125.3490	0.065554	15.6536	9.1126
AC-20%	667.8184	564.8156	0.296032	93.3126	9.6193
AC-30%	731.8978	627.4067	0.328940	92.0292	9.7589
AC-50%	756.3981	569.7129	0.301339	93.0393	9.9813

4.2.2 Effect of acid concentration on specific surface area

The result showed (Table 3) that initially the surface area for the raw material was 0.6598 m²/g. Figure 2 illustrates that after the raw material were impregnated with acid phosphoric with different concentration, all the activated carbon showed drastically changes in the surface area and extensively produced higher surface area than the raw mangosteen. The results proved that chemical activation offer advantages by forming higher surface area.

The activated carbon prepared with 10% acid concentration has 143.7346 m²/g and the activated carbon prepared with 20% acid concentration has 667.8184 m²/g. The 20% acid concentration produced activated carbon with four folders higher in term of surface area than the 10% acid concentration. Besides that, the activated carbon with 10% concentration (143.7346 m²/g) was not in the range of activated carbon surface area. Typically, the surface area of an activated carbon ranged from 400 – 1500 m²g⁻¹ (Knozinger *et al.*, 1999).The activated carbon with 30% and 50% produced activated carbon with 731.8978 m²/g and 756.3981 m²/g respectively.

Apart from that, when the chemical ratio increase the average pore diameter of the prepared activated carbon were increase from 9.1-9.9A (Table 3). This is due to the strong inhibition of tar formation by H_3PO_4 which promotes release of volatiles from the carbon structure and contributes to substantial increase in widening of pores.

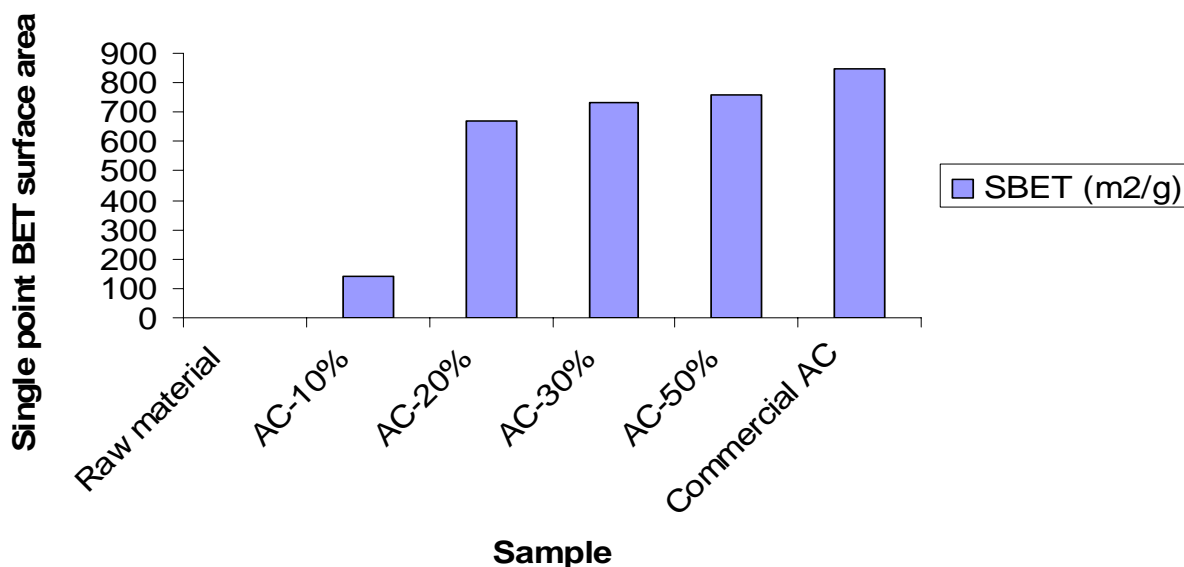


Figure 2: Single point BET of raw material, commercial AC and prepared AC

4.2.3 Effect of acid concentration on pore structure

The shape of the adsorption isotherms can provide qualitative information on the adsorption process and the extent of the surface area available to adsorbate. Nitrogen adsorption isotherms reported in Figure 2 illustrates that similar shaped isotherms (Type 1) were also obtained for samples with different impregnation ratio/acid concentration. This indicated the existence of microporous structure were pore filling which occurred at low relative pressures. An absence of hysteresis indicated the absence of mesoporosity and suggested that the carbon products contained mostly micropores with only a small contribution of mesopores. Raising the amount of acid phosphoric led to loss in micropore which could be due to the collapse of micropore walls and resulting in pore widening and an increase in mesopores portion (P.Patnukao and P.Pavasant, 2008). This is clearly illustrated in Table 3 where the micropore decreased along as a result of an increase in the acid content during activation process. The average pore diameter for all prepared activated carbon were in range 9.1-9.9A which were classified as micropores by the International Union of Pure and Applied Chemistry (IUPAC) due to the diameter which are less than $<2\text{nm}$. Therefore, in this study, 30% acid phosphoric content produced activated carbon with high surface area and good pore size distribution which mainly are micropores.

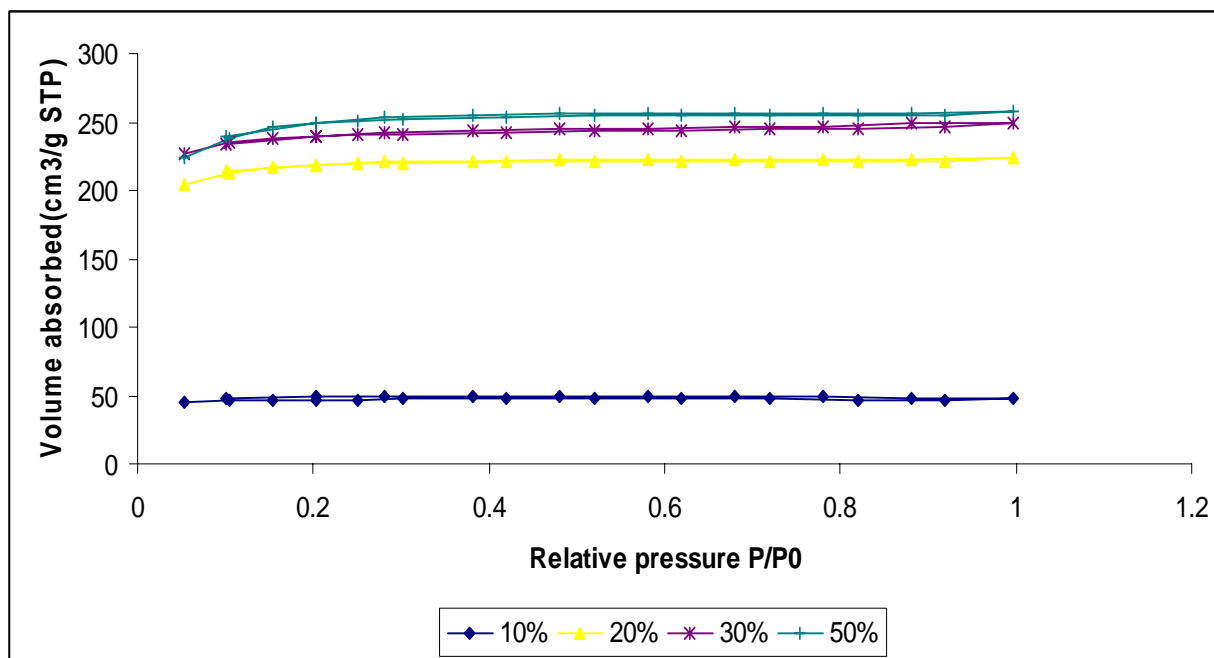


Figure 3: Adsorption isotherms of N₂ at 77.30K on activated carbon prepared at different impregnation ratios

5.0 Significance of finding

Mangosteen peel can be used as precursor for the activated carbon production due to high carbon content and low ash content. Mainly, all the activated carbon prepared with different acid phosphoric from mangosteen consists of micropores and the 30% acid concentrations are the most suitable for activated carbon production.

6.0 References:

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