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Preparation of activated carbon from Phu Phan Dendrocalamus asper backer

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Abstract

The abstract reveal the objectives of the research and the results proposed: the objective of this research is to prepare activated carbon from Phu Phan Dendrocalamus asper backer obtained from Phu Phan district Sakon Nakhon province. The effect of chemical activation process of activated carbon was immersed in a 35% hydrochloric acid solution. The physical properties such as moisture, ash, volatile and fixed carbon contents were measured. The results were found that the optimum carbonization temperature was 550 ˚C for 2 h. The amount of ash and fixed carbon were 0.35% and 97%. It was then activated by microwave radiation at 800 watts for 60 sec. Finally, it was ground and sieved with a sieve shaker (size 75 µm). The surface area, total pore volume and pore radius were obtained by 20.47 m^2/g, 0.0467 cm^3/g, and 4.56 nm, respectively. The crystal structure was analyzed by X-ray diffraction and the peak positions were confirmed at the corner (14˚, 24˚, 26˚, and 29˚). The results showed that the activated carbon had a graphite crystal structure, whereas the peak in the position 30˚-50˚ was low, indicating that the structure was contaminated with amorphous carbon. An explanation for this is that during the heating process, the arrangement of carbon is the plane of graphite, and during carbonization, there is a random distribution of graphite structures, resulting in a lack of integrity in the carbon crystal structure. The average size according to scanning electron microscopy was between 60-240 µm and the Vicker hardness was 92.

Keywords: Activated carbon, Fixed carbon yield, HCL, Phu Phan Dendrocalamus asper backer

1. Introduction

Thailand has experienced rapid industrial expansion. The industry groups that have had the highest continuous growth include textiles, food, steel, automobiles and electricity and electronics. Meanwhile, these industries have also affected the environment, particularly water pollution from chemical contaminants such as organic and inorganic matters, which have an impact on natural water sources [1]. Natural water sources are very important in the countryside for living, consumption, cultivation and livestock. Therefore, the wastewater produced from manufacturing industries must be treated before releasing it into natural water sources to prevent and control organic and inorganic matter contamination. There are many methods for industrial wastewater treatment to reduce the amount of organic and inorganic matter. An absorption technique is a popular wastewater treatment method with ongoing research due to several advantages, including a relatively low absorbent production cost and a short treatment time. In addition, it can be operated under normal temperature

and pressure. Moreover, it does not require chemicals and only uses activated carbon absorbents [2]. Activated carbon is, essentially, a carbon-based material that can be produced from a number of carbon-rich organic materials including biomass, biomass cellulose, peat, lignite and coal [3]. Several studies have investigated the production of activated carbon from agricultural residues due to their low cost and environmentally friendly nature, such as apricot shell, corncob, coconut husk, jackfruit peel, olive tree wood, olive stones, banana empty fruit bunch, oil palm shell, hard wood, date stems, marigold straw, rice and coffee husk [4-14]. There are two different methods for manufacturing activated carbon from agricultural wastes-physical and chemical activations. Physical activation involves carbonization of precursor followed by activation of the resulting char in presence of activating agents such as CO2 or steam. For chemical methods, first impregnation of precursor with the selected chemical followed by carbonization and activation [15]. In case of acid process, charring of the material done with acid such as HCl followed by activation. Chemical activation is a widely used method for preparing activated carbon due to its simple technique, short preparation time, low temperature of activation, high surface and porosity. Due to the numerous small pores spread over a large surface, it has a large surface area with high absorption capacity to absorb the organic contaminant in wastewater as catalyst supported. The synthesis of activated carbon can be accomplished by stimulation to increase the absorption capacity, surface area and porosity. Bamboo is a tropical plant commonly found in Thailand, China and Vietnam. Sakon Nakhon is one of the northeastern provinces of Thailand. It is located on the Khorat Plateau [16]. Phu Phan Dendrocalamus asper backer bamboo is encouraged to be planted in Sakon Nakhon Province, Thailand owing to its medium-size bamboo. The characteristics of Phu Phan Dendrocalamus asper backer bamboo were reported (the stem as the dark green) with the diameter is about 9-12 cm. The circumference is in the range of 30 - 40 cm. The stem is not more than 10 m, with roughness of the stem surface and has a high amount of organic carbon, which could increase economic value when using it to produce activated carbon without having to destroy trees. Therefore, the objective of this research is to prepare an activated carbon from Phu Phan Dendrocalamus asper backer to develop and created additional value by producing activated carbon using this material as an activated carbon for maximum benefit. The physical properties including moisture, ash, volatile and fixed carbon contents were analyzed.

2. Materials and methods

2.1 Activated carbon preparation

Phu Phan Dendrocalamus asper backer was washed with clean water, and then dried by sunlight for approximately 1 week, finally weighing 10 kilograms. It was carbonized in gasifier between 600-750 ˚C for 2 h, resulting in the activated carbon. The material was then crushed in a ball mill (Retsch PM400 MA) for 30 minutes and sieved with a sieve shaker, size 75 inches (Retsch, A5200-Basic.) The material with the most fixed carbon content was soaked with 40% hydrochloric acid solution at the ratio of material 1 g/20 mL. Hydrochloric acid (HCl) of analytical grade was purchased from Merck. Then physically treated by wave energy (micro wave frequency 800 watts) for 60 sec. Moisture content of Phu Phan Dendrocalamus asper backer powder was analyzed, according to ASTM D3173-95 [17] and was achieved by baking at 150 ˚C for 2 h in a ceramic cup closed with a lid, which uses a microwave oven (EMM20D, Electrolux, China). The samples were placed in a desiccator to reduce the temperature and then weighed. Finally, a 1 g sample was weighed in a cup with a lid and baked at 150 ˚C for 10 h, followed by cooling to room temperature and weighing.

2.2 Characterizations

2.2.1 Stephen Brunauer, Paul Hugh Emmett and Edward Teller (BET)

The theory aims to explain the physical adsorption of gas molecules on a solid surface and serves as the basis for an important analysis technique for the measurement of the specific surface area of materials. The percentage of moisture content is calculated using Equation (1):

$$\text{Percentage of moisture content} = \frac{(C-D)}{(C-B)} x100 \quad (1)$$

Where B = weight of a ceramic cup with a lid (g)
C = weight of the ceramic cup and lid combined with the sample weight before baking (g)
D = weight of the ceramic cup and lid combined with the sample weight after baking (g)

Volatile substance content was analyzed according to ASTM D5832-98. The percentage of moisture content was calculated using Equation 2:

$$\text{Percentage of weight loss} = \frac{(C-D)}{(C-B)}x100 \quad (2)$$

Equation 3 is used for calculation of percentage of volatile matter by the relationship with Equations (1) and (2) as presented below:

$$\text{Percentage of volatile matter} = (2) - (1) \quad (3)$$

Ash content was analyzed according to ASTM D2866-11. The percentage of moisture content was calculated from Equation 4:

$$\text{Percentage of ash content} = \frac{(C-B)}{(C-B)}x100 \quad (4)$$

The fixed carbon content was analyzed according to ASTMD 3172. The remaining product from burning after removal of moisture, volatile matter and ash can be calculated from Equation (5):

$$\text{Percentage of fixed carbon} = 100 - (M + V + A) \quad (5)$$

Where M = percentage of moisture
V = percentage of volatile matter
A = percentage of ash

The surface area and porous volume of the activated carbon was analyzed with the BET technique [18]. This technique uses the Auto sorb 1-MP from Quanta chrome, which absorbs via nitrogen gas (N_2) under standard pressure at 150 ˚C. The amount of absorbed gas (W) and the relative pressure (P/P_0) of the added material are applied in the following equation called the "BET equation":

$$\frac{1}{w\left[\frac{P_0}{P}-1\right]} = \frac{1}{cW_m} - \frac{c-1}{cW_m}\left(\frac{P}{P_0}\right) \quad (6)$$

Where W = amount of N_2 absorbed at the relative pressure P/P_0
W_m = amount of N_2 that is absorbed on the surface of the substance in a single molecule
P = pressure of N_2 used during the experiment (mHg)
P_0 = saturation pressure of N_2, used during the experiment (mmHg)
$C =$ constant that depends on the energy to absorb (26.36)

From the BET relationship equation when the graph is plotted between

$$\frac{1}{w\left[\frac{P_0}{P}-1\right]} \text{ and } \frac{P}{P_0}$$

A straight line graph is obtained with a slope (S) of: $Y = aX + b$

$$\frac{\mathrm{P}}{w[\mathrm{P_0}-\mathrm{P}]} = \frac{\mathrm{P}}{P_0}\left(\frac{C-1}{cW_m}\right) + \frac{1}{cW_m} \quad (7)$$

And a Y axis intercept (i) of

$$i = \frac{1}{cW_m} \quad (8)$$

The amount of N_2 absorbed on the surface of a single molecule (W_m) is calculated by applying S and I in Equation 9:

$$W_m = \frac{1}{S+i} \quad (9)$$

The specific surface area of the material is calculated by using W_m in equation 10:

$$S_t = \frac{W_m N A_{CS}}{M} \quad (10)$$

Where S_t = surface area of the material (m^2)

N = Avogadro's number (6.023×10^{23}) (molecules per mol)

M = molecular weight of N_2 (28 g/mol)

A_{cs} = cross-sectional area of the N_2 molecules that are absorbed (16.20×10^{-23}) (m^2)

S_t = surface area is divided by the amount of the sample material used for the list test (W), yielding square meters per gram:

$$S = \frac{S_t}{W} \tag{11}$$

The total porous volume (V_p) and the average pore size (r_p) of the sample material can be calculated from the relationship of the equation as follows:

$$Vp = \frac{W_s}{P}$$
$$\overline{rp} = \frac{2Vp}{S_t} \tag{12}$$

Where W_s = amount of N_2 absorbed on the surface of the sample material at a relative pressure ($P/P_0 \approx 1$)

p = density of N_2 absorbed on the surface of the sample material at a relative pressure ($P/P_0 \approx 1$)

S_t = surface area of the tested sample material

2.2.2 X-ray diffraction techniques

The X-ray diffraction technique (XRD) was applied to analyze its crystal activated carbon powder using angles between 14 and 46 degrees on a Shimadzu X-ray diffractometer 6100. The angle can be calculated from the Bragg equation as follows (13):

$$\sin\theta = \frac{QS}{d} = \frac{SP}{d}$$
$$d\sin\theta = QS = SP$$
$$2d\sin\theta = QS + SP$$

but $QS + SP = n\lambda$

then $2d\sin\theta = n\lambda$ (13)

where λ is the wavelength of radiation; n is the order of reflection; and d is the plane distance

Next, 5 g of activated carbon powder was extruded with a vacuum heat press (serial no.OTF-1200X - VHP4 OTF-1200X-VHP4) at 800 ˚C for 3 h. The activated carbon lump was cut with a low-speed cutting device (serial no. Isomet 1000) with a precision size of 3×3×15 mm^3. The activated carbon lump was scrubbed with a polishing machine (MARUTO, Doctor Lap).

2.2.3 Scanning electron microscopy surface microstructure

The morphology and surface area characteristics of the activated charcoal granule were investigated using scanning electron microscopy (SEM) (Hitachi, TM4000Plus). Prior to the analysis, the samples were attached to the sample holder with carbon taped and covered with gold to make them conductive before analysis.

2.2.4 Hardness

The hardness was measured using a Vickers hardness tester (HMV-2, SHIMAD ZU) as a physical property. The hardness value can be calculated according to the following Equation (14):

$$HV = \frac{2F\ \sin\frac{136°}{2}}{d^2}$$
$$HV = 0.1891\frac{F}{d^2} \tag{14}$$

Where HV is the Vickers hardness; F is the weight used to press; and d is the diagonal length of the pressing.

3. Results and discussion

The activated carbon granules were synthesized from Phu Phan Dendrocalamus asper backer from the Phu Phan district, Sakon Nakhon province according to ASTM standards. The physical and chemical properties of activated carbon were analyzed including surface area, porous volume, crystal structure, microstructure and hardness. The results revealed that the contents of moisture, ash, and volatile were low whereas the fixed carbon content was high, resulting in the yield of activated carbon produced. This was implied that the activated carbon obtained was influent from the fixed carbon content of the material used. The material with low fixed carbon content but high volatile content produced in less activated carbon.

The preparation of Activated Carbon from Phu Phan Dendrocalamus asper backer by using microwave radiation was 1.37% of moisture content, 0.85% of volatile matter, 0.30% of ash content and 97.06% of fixed carbon content as reported in Table 1. Activated Carbon from Phu Phan Dendrocalamus asper backer synthesized by this study was demonstrated that the contents of moisture, ash, and volatile were low, in contrast, the fixed carbon was high. This result was similar with the study of Chotitham, *et. al.* [19] that fixed carbon was more than 80% but volatile matter was less than 10%. Phu Phan Dendrocalamus asper backer, therefore, was suitable for preparation as an activated carbon due to high yield product. Moreover, hydrochloric acid (HCl) solution is an important role to remove the volatile matter in activated carbon, leading to high fixed carbon and low volatile matter. According to the study of Ademiluyi and David-West [20] reported that the activated carbon from bamboo with HCl as an activating agent for heavy metals adsorption presented that the activated carbon from bamboo using HCl gave the 2nd high iodine number after HNO_3 but it was better than H_2SO_4, H_3PO_3, NaOH and $ZnCl_2$. The heavy metal adsorptions by using HCl gave high efficiency as well as HNO_3.

After bamboo activated with HCl, a highly reactive product known as cellulose chloride that possible make the HCl a reactive activating agent as follows:

$$C_6H_{10}O_5 + 5HCl \rightarrow 5H_2O + C_6H_5Cl_5 \quad (15)$$

Table 1 The average proximate properties of activated carbon of Phu Phan Dendrocalamus asper backer.

Temperature (˚C)	Moisture content (%)	Volatile matter (%)	Ash (%)	Fixed carbon (%)
550	1.37	0.85	0.30	97.60
650	2.02	1.19	0.39	96.35
750	1.50	0.99	0.36	97.06

The optimum temperature for the 2 h carbonization of Phu Phan Dendrocalamus asper backer varied (550, 650, and 750 ˚C) obtained is shown in Figure 1. In addition, the temperature of carbonization per fixed carbon percentage and product percentage was found to be 550, 650, and 750 ˚C for 2 h, which provided a fixed carbon content of 97.60%, 97.06% and 96.35%, respectively, and product percentages of 28.87%, 23.67% and 21.62%, respectively. These results are agreeable with Mahanim, *et al.* [21] presenting that the fixed carbon content in samples changes when exposed to high temperatures during carbonization and activation.

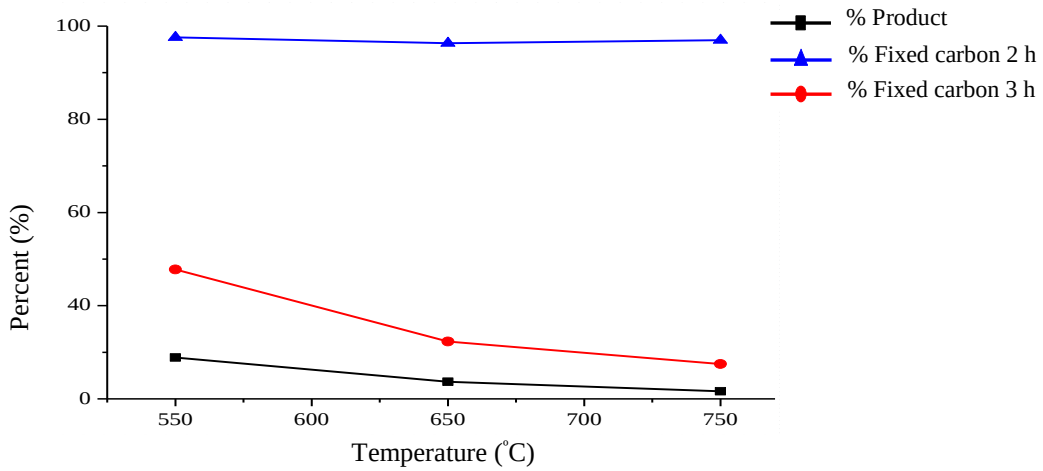

Figure 1 Carbonization at different temperatures.

The crystallographic structure of the activated carbon powder was characterized by Shimadzu XRD 6100 at angles between 5 and 80 degrees. The diffraction patterns were run with copper radiation (λ=0.15405 nm) at 40 KV and 30 mA, and scanning speed of 2 degree/min. The XRD spectrum in scale is show in Figure 2. As shown in Figure 2, the activated carbon from bamboo has indicated by dominant peaks at 2θ of 24.36°, 43.45° and 45.48°, corresponding to (111), (100) and (110) hexagonal crystal planes, respectively. Moreover, there are new peaks formed at 14.87, 20.49, 26.58, 29.25, and 30.06 corresponding to monoclinic crystal planes of diphenyl acetylene, respectively. This result showed that carbonization of bamboo between 550 ˚C to 750 ˚C didn't transform the cellulose to carbon completely. Nonetheless, quantification of the two phases revealed that more than 60% of the cellulose has been transformed into carbon, leading to select for further testing.

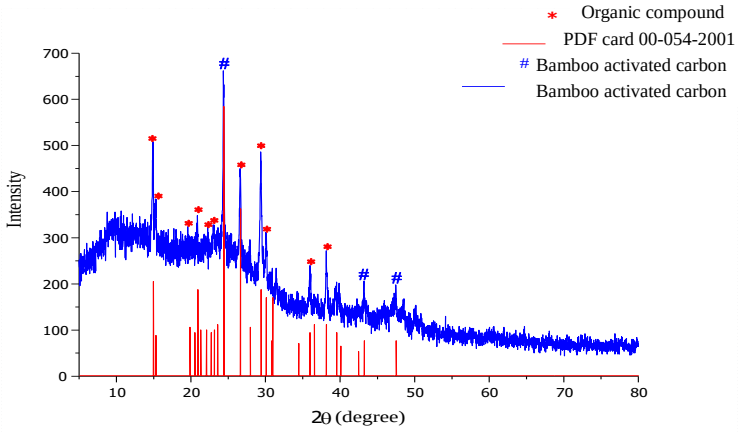

Figure 2 The crystal structure analysis of activated carbon powder by X-ray.

3.1 Surface morphology of activated carbon

The activated carbon samples produced from Phu Phan Dendrocalamus asper backer were characterized by SEM in order to determine the diameter of vascular bundles of activated carbons employed. Preparation of activated carbon based Phu Phan Dendrocalamus asper backer largely depended on the porous size and distribution of porous materials. The images of SEM were illustrated in Figure 3, performed at different magnifications of 250x, 500x, 1,000x and 3,000x. The average pore size and the diameter were 60 µm and 240 µm, respectively, the results found that the activated carbon obtained by our method gave the uniformed mesoporous and microporous. The results of SEM found that there are holes and cave type opening on the surface of the specimen that would have increased surface available for adsorption. The surface area of activated carbon would enhance by the presence of more porosity [15].

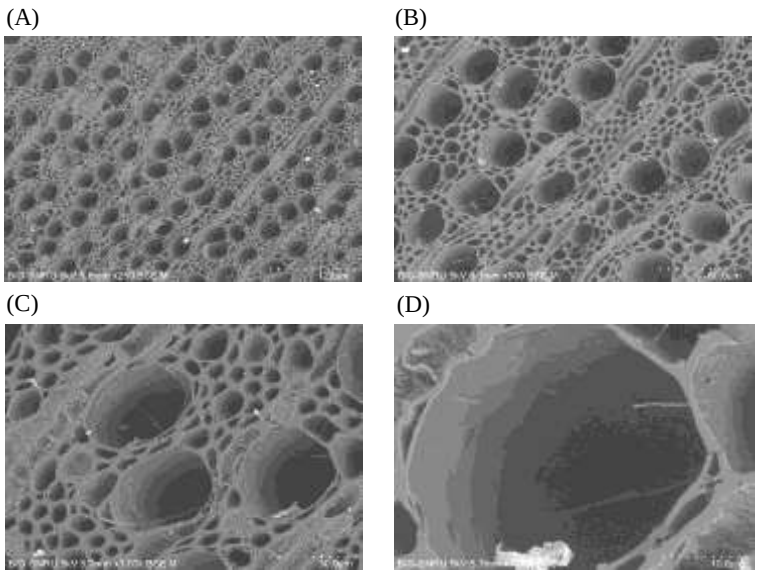

Figure 3 SEM activated carbon (A) at 250X (B) at 500X (C) at 1000X and (D) at 3000X**.**

3.2 Isotherm of Phu Phan Dendrocalamus asper backer

According to the adsorption isotherms of N_2, the experiment was done to analyze the surface area and the total pore volume of Phu Phan Dendrocalamus asper backer by using BET. The surface area and the total pore volume of activated carbon were 20.47 m^2/g and 0.0467 cm^3/g, respectively, as shown in Table 2. The average pore size was 4.56 nm which was suggested that the activated carbon obtained was categorized as the adsorbent with mesoporous material.

According to the IUPAC, it is known that in a porous material there must be a compromise between diameter pore size of micropore and mesopore. There were 3 different groups of adsorbents using diameter of pore size which were micropore (< 2 nm), mesopore (2-50 nm) and macropore (> 50 nm). Based on this work, only experimental evidences of micropore in activated carbon synthesized from Phu Phan Dendrocalamus asper backer.

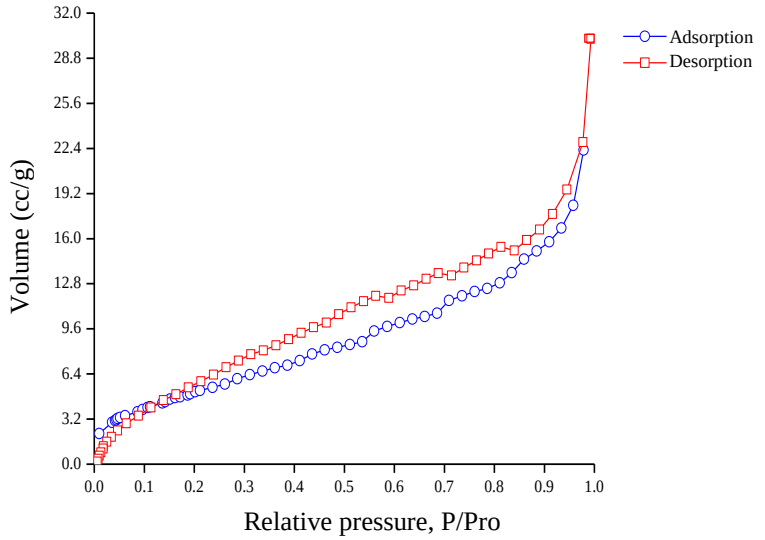

Figure 4 Volume of N_2 adsorption isotherm versus relative pressure in activated carbon obtained from Phu Phan Dendrocalamus asper backer.

3.3 Surface area and pore size analysis

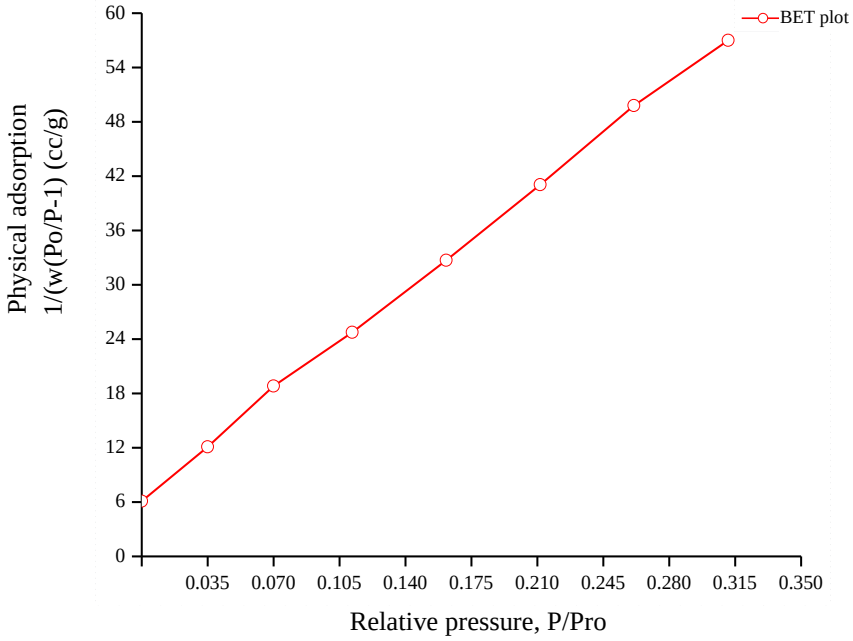

Figure 5 Surface area analysis of activated carbon (75 µm).

Figure 5 shows the relationship between the BET equation of the amount of absorbed gas (W) and the relative pressure (P/P_0) of the increased material. This relationship can be used to calculate the specific surface area (as shown in Table 2).

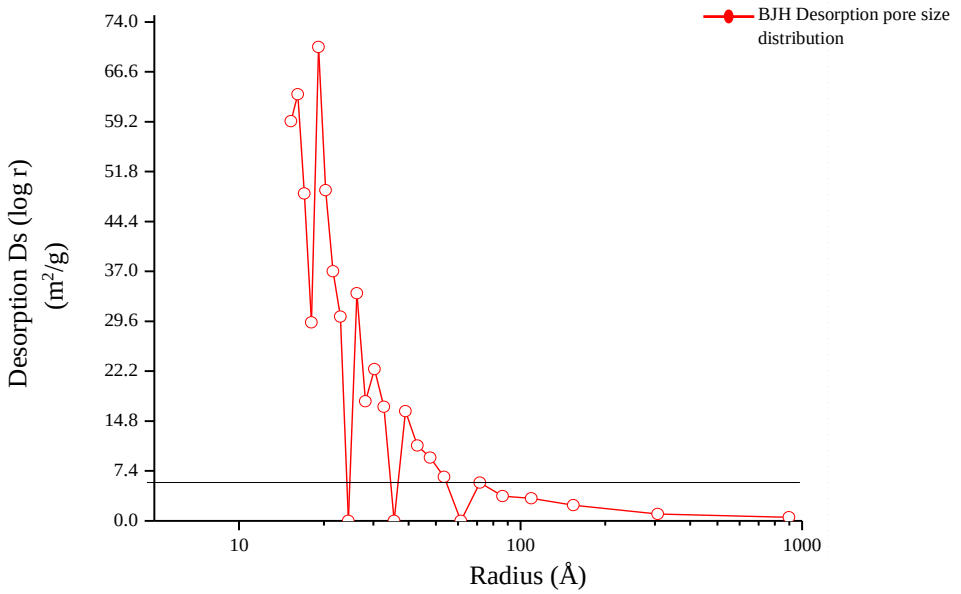

Figure 6 Results of the pore size analysis.

The analysis of the surface area and the pore size found that the activated carbon granules were 75 µm in size, with a specific surface area of 20.47 m^2/g and a porous volume of 0.0467 cm^3/g. The pore size of the activated carbon had a radius of 4.56 nm and a small radius of 1368.1 nm. Therefore, the activated carbon granules have a very porous volume, resulting in a large specific surface area. The properties of this activated charcoal endow it with a large ion-storage capacity.

The activated carbon granules (size 3x3x15 mm^3) had an average Vicker hardness (HV of approximately 92). The hardness value represents a material's stability and its ability to resist corrosion.

The result shows that the hardness value of activated carbon granules is in the Thai industrial standard of activated carbon, which must be more than 70% [22].

Table 2 Specific surface area and pore size analysis of activated carbon granule.

Activated carbon properties	Activated carbon granule (size 75 µm)
Specific surface area, S(m^2/g) BET theory	20.47
BET C-constant	26.36
Volume of pore (cm^3/g)	0.0467
Average pore size radius (nm)	4.56
Pore size with small radius (nm)	1368.10
Reference document	S62113AC.RPT

4. Conclusion

The optimum conditions for the synthesis of activated carbon from Phu Phan Dendrocalamus asper backer were investigated by both chemical stimulation, using oxidizing agents, and by physical stimulation, using microwave frequency energy, in the carbonization process at a temperature of 550-750 ˚C for 2 h. The optimum carbonization temperature was 550 ˚C, which yielded 97.60% fixed carbon content and 28.87% product content. The crystal structure analysis using XRD found that the material was a high-carbon compound compared with international reference standards. According to the XRD JCPDS card 00-054-2001, it had a hexagonal crystal structure. SEM analysis revealed that the microstructure of the granule had a small porosity and even distribution. The average large pore size was 60 µm. The diameter of the small pore size was 240µm. In addition, small porosity was placed inside the holes of large porosity. The results of the activated carbon granules (75 µm) had a specific surface area of 20.47 m^2/g and a porous volume of 0.0467 cm^3/g. The activated carbon granules (size 3x3x15 mm^3) had an average Vicker hardness of approximately 92. When carbonizing the

biomass materials at temperatures below 400 ˚C, the volatile matter in the biomass material did not decay into fixed carbon. However, above 750 ˚C, the reaction caused the carbon to turn into a gas, resulting in a decrease in the amount of activated carbon. The activated carbon obtained from Phu Phan Dendrocalamus asper backer can be used as an alternative absorbent material.

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