Effect Of Activation Temperature And Time In The Development Of Activated Carbon Derived From Banana Peel

by

Muhammad Hambali bin Nazarudin

14567

Dissertation submitted in partial fulfillment of

the requirement for the

Bachelor of Engineering (Hons.)

(CHEMICAL ENGINEERING)

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CERTIFICATION OF APPROVAL

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Approved by,

____

(Mr.Azry Borhan)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD HAMBALI BIN NAZARUDIN

ABSTRACT

The main aim of this project is to study the prospective of using banana peel as potential adsorbent in eliminating carbon dioxide from polluted air in the atmosphere. The study emphasizes on the parameters involved in the preparation of the banana peel biosorbent, such as activating agent, activation temperature and activation time. Initially, the dried banana peel is grinded into powder form, then chemically activated using either potassium hydroxide and carbonized at different sets of temperature and time. This carbonization process allows the formation of activated carbon, one of a common type of adsorbent used globally especially in industries' effluent treating. The biosorbent then is further heated by microwave-heating method in order to find out either the product becomes better or vice versa. The characterization of agro-based activated carbon is done using the surface area analyzer (Micromeritics ASAP 2020) and the Field Emission Scanning Electron Microscope (FESEM) to determine which set of parameters produces the largest surface area estimated using the BET theory (S_{BET}) which is directly proportional to the effectiveness of biosorbent. In addition, the activated carbons developed from banana peel are also tested for their adsorption capacity with typical pollutants found in industrial emission such as carbon dioxide using HPVA II analyzer. The samples were also analysed for its elemental composition before and after carbonization which results in 58.67 wt% and 56.18 wt% respectively for its carbon content. Sample MW2 was recognized to yield activated carbon with largest surface area, S_{BET} of 1142.7893 m^2/g and pore diameter of 2.22765 nm which falls in mesoporosity subclass. Sample MW2 with particle size of 0.25 mm was prepared using potassium hydroxide as activating agent with 1:1 impregnation ratio and carbonized for two hours at 400^oC. Sample MW2 then is further tested for carbon dioxide adsorption at 25^oC which is able to adsorb carbon dioxide.

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A very special note of thanks to my supervisor, Mr.Azry Borhan, who was always willing to assist me and provided good support throughout the project completion. His excellent support, patience and effective guidance brought a great impact to my project as well as me.

Nevertheless, I would like to thank the FYP committee for arranging various seminars as support and knowledge to assist the students in the project. The seminars were indeed very helpful and insightful to me. I would also like to thank all lecturers from Universiti Teknologi PETRONAS who had given me guidance throughout the period of the project.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

This project is related to the removal of carbon dioxide from gas/air streams focusing in adsorption by chemical activated carbon by potassium hydroxide extracted from banana peel wastes then further enhancement by microwave-heating method. Currently, there are several methods that are used commercially in the process of carbon dioxide removal which includes membrane separation, chemical conversion and cryogenic separation as well as by adsorption both on physical and chemical on a solid surface (Ning et al., 2012). Contemporary researchers are studying the advantages of using by-products and agricultural wastes such as banana peel into activated carbon (Djilani et al., 2012). The ultimate aim of this research is to develop activated carbon from banana peel and eventually provide a suitable platform for adsorption of carbon dioxide to take place. This study also could investigate the effectiveness of the activated carbon in removing carbon dioxide. Activation of raw materials produces porous structure within these materials, which allows for remarkable adsorption capacities. Basically, there are two methods used in carbon activation, namely the physical activation and the chemical activation (Djilani et al., 2012). The parameters that will be studied in this experiment are chemical activation with the optimum activation temperature, optimum activation time, pore characteristics of activated carbon and carbon dioxide adsorption capacity.

1.2 Problem Statement

Lately, the environmental effects of carbon dioxide are very significant. Carbon dioxide is an important greenhouse gas that is produced from burning of hydrocarbons since the industrial revolution has rapidly increased the concentration, leading to global warming. Since 1751 approximately 337 billion metric tonnes of carbon have been released to the atmosphere from the consumption of fossil fuels and cement production (Marland, Boden, Andres, Brenkert, & Johnston, 2003). According to statistical survey by Wikipedia, in the year 2008, China holds the first place in emitting CO_2 with annual emission of 7,031,916, 000 tonnes followed by United Stated with 18.27% of world emission, that is 5,461,014, 000 tonnes. The world annual emission in 2008 is 29,888,121, 000 tonnes. In conjunction with developing eco-friendly technology to reduce the carbon dioxide emissions, a lot of researches have been done with different experimental methodology to define the best way on carbon capture. One of the most economically and environmentally preferred technology is adsorption by agriculture wastes. Due to its high potential on adsorption even before carbonization, banana peel would have large surface area and porosity as an activated carbon is excellent adsorbent and widely used for carbon dioxide removal (Jang & Park, 2012).

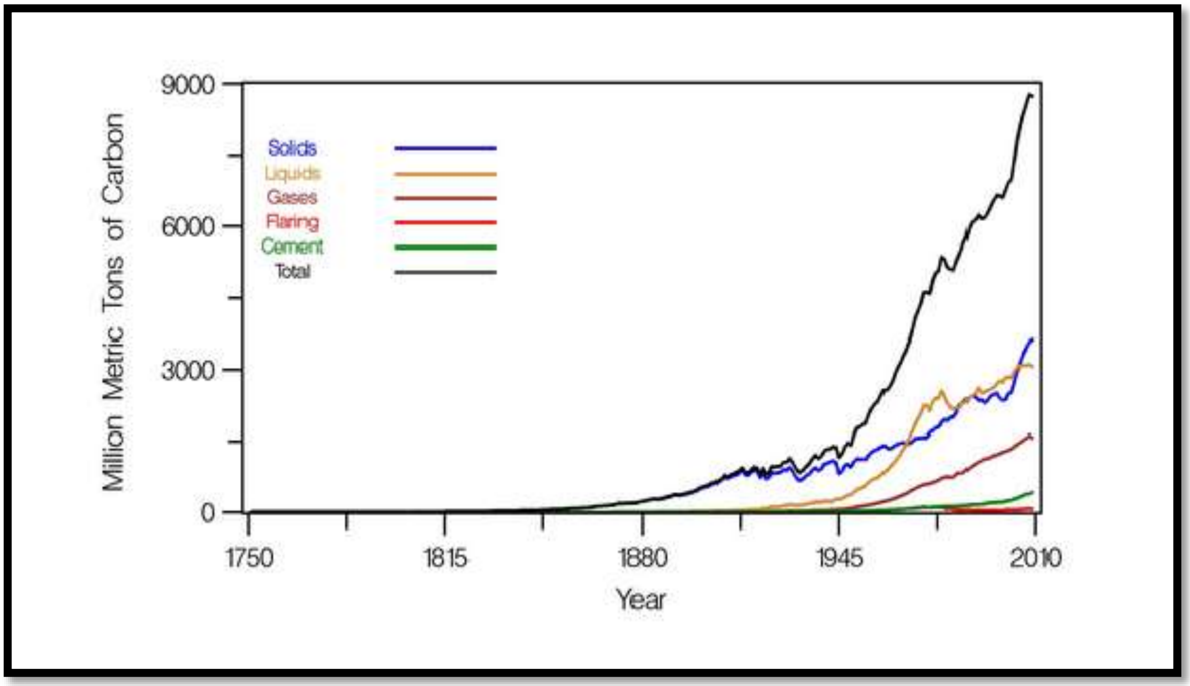

Figure 1 : Carbon Emission Estimate from 1751 until 2010 (Marland, Boden, Andres, Brenkert, & Johnston, 2003)

1.3 Objectives of Study

Three objectives were highlighted in this study :

- Preparation of activated carbon derived from banana peel by chemical activation using conventional-heating method then further heated by microwave-heating method
- Characterization of pores of activated carbon by studying their pore size and adsorption isotherms accordingly

- Utilization of banana peel for carbon dioxide adsorption by studying the comparison of adsorption capacity using two different gases, carbon dioxide and nitrogen

1.4 Scope of Study

The experiment will use potassium hydroxide (KOH) as activation agent to activate carbon content in banana peel. Activated carbon will then be further heated with microwave-heating method. The pores of activated carbon are then tested for their characteristics with nitrogen (N_2) gas. The experiment is repeated for different operating parameters:

1. Activation temperature

2. Activation time

CHAPTER 2

LITERATURE REVIEW

2.1 Adsorption

Adsorption is defined as the process of the adhesion of one or more component of a gas or liquid stream on the surface of a solid adsorbent to accomplish a separation process. The film of components in which adsorbed to the adsorbent surface is known as the adsorbate(Geankoplis, 2003). The process continues until equilibrium is established between the amount of solid-bound adsorbate and the remaining portion in the solution. Adsorption can be categorized in to two types, namely physisorption and chemisorption. Physisorption typically involves weaker bonding forces among the molecules (e.g. Van der Waals force). It usually occurs between the adsorbed molecules and the adsorbent internal pore surface. Another trait of physisorption is that it is readily reversible, which means that desorption can be achieved easier. On the other hand, chemisorption usually involves strong chemical bonding (e.g. covalent bond). The process is also more selective and highly specific on the adsorbate molecules and it usually occurs in monolayer (Geankoplis, 2003).

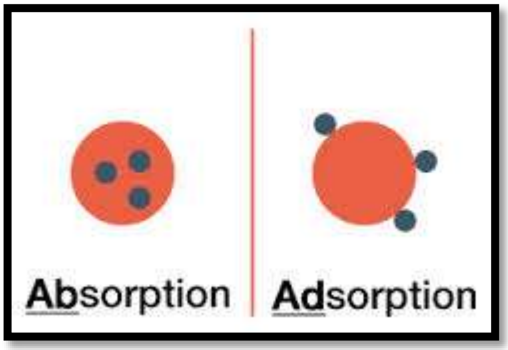

Figure 2 : An overview diagram on difference between absorption and adsorption

2.2 Activated Carbons

Activated carbon is charcoal that has been treated with oxygen to open up millions of tiny pores between the carbon atoms ("What is activated charcoal and why is it used in filters?", 2000). Activated carbon has a tremendous adsorptive capacity, an affinity for a wide variety of dissolved organics and chlorine and an ability to be custom-tailored to suit specific applications. The carbon-based material is converted to activated carbon by thermal decomposition in a furnace using a controlled atmosphere and heat. The resultant product has an incredibly large surface area per unit volume, and a network of submicroscopic pores where adsorption takes place. Amazingly, one pound of carbon provides a surface area equivalent to six football fields (Jang & Park, 2012).

Figure 3 : Activated carbon structure under electronic microscope magnification

2.3 Banana Peels

A banana peel is the outer skin layer which covers the soft yellowish banana fruit. Bananas gained its popularity worldwide with yearly production over 145 million tonnes in the year 2011. With regard to its consumption, there is a significant amount of banana peel waste being generated as well. According to the Survey Report on the Distribution and Waste Disposal of Bananas by the Association of Australian Banana Wholesalers 2006, 68.6 tonnes of bananas collected as raw material waste annually. The banana waste disposed at municipal tip or collected as organic waste was 87%. On average, banana peels contain 6-9% dry matter of protein and 20-30% fibre. Other than that, the elemental analysis of af a banana peel show that it have a very high carbon content to be used as activated carbon which is 78.41(wt%)(El-Nafaty, Muhammad, & Abdulsalam, 2013). On the other hand, in Malaysia, there is 29,270 hectare of banana planted in the year 2012 producing 294,530 metric ton of fresh banana(Anem, 2012). The total value of banana production was estimated about RM 294.5 million in the same year. In addition, the most common type of banana cultivated in Malaysia is the Cavendish type. Banana peels are also used for water purification, and ethanol, cellulose as well as laccase production. This agricultural waste material is composed mainly of lignin and cellulose(Deithorn & Mazoni, 2014). Cellulose can be used as an adsorbent for the carboxyl and hydroxyl functional group which becomes the active binding site of the metal (Deithorn & Mazoni, 2014). Other components include hemicellulose, extractives, lipids, proteins, simple sugars, starches, water, hydrocarbons, ash, and other components with numerous functional groups. Besides that, these components contained in banana peel facilitate the metal complexations which directly aid the sequestering of heavy metals as well as exhibit metal biosorption capacity (Deithorn & Mazoni, 2014).

Figure 4 : Pile of banana peel waste

2.4 Activation Methods

Activated carbon can be manufactured from a variety of precursors via physical or chemical activation. Physical activation is usually carried out in two steps: firstly, carbonization of raw material is done in an inert atmosphere at a temperature below 700°C, and the second step is the activation in the presence of steam, carbon dioxide, and/or air at temperatures between 800 and 1000°C. Chemical activation is generally carried out in one step, which consists of the impregnation of the raw material with a strong dehydrating agent, such as H_3PO_4, $ZnCl_2$, K_2CO_3, NaOH or KOH, and then heating the mixture to temperatures of 400-800°C to simultaneously form and activate the carbon(Marsh & Rodriguez-Reinoso, 2006). Chemical activation provides a few advantages over physical activation which include single step activation, low activation temperatures, shorter activation time, higher yields and better porous structure. The chemical agents used are usually substances with dehydrogenation properties that inhibit the formation of tar and reduce the production of other volatile products. The disadvantage of chemical activation process is the need for an important washing step, which is time consuming due to number of washings required to completely remove the activation agent from the carbon(Lim, Srinivasakannan, & Balasubramaniam, 2010).

2.5 Microwave-Heating Method

Commonly, the conventional-heating method would be using furnace in order to achieve hight temperature to activate the carbonaceous material but it will take a long time. Rather than using high temperature for a long time could use up a lot of energy and recently researchers are diverting into using microwave-heating method for shorter time. Among these methods, adsorption processes have become increasingly competitive and already favorable because of its low energy requirement, easy operation, and low maintenance(Pires, Bestilleiro, Pinto, & Gil, 2008),(Xu, Zhoa, Sun, & Liu, 2009). It is well known that an excellent adsorbent is key to adsorption separation of the CO2 system. Various materials such as zeolites(Llano-Restrepo, 2010),(Delgado & Arean, 2011), activated carbons(Arami-Niya, W. Daud, & S., 2011),(Aroua, W. Daud, Chun, & Adinata, 2008), and carbon molecular sieves(Adinata,

W. Daud, & Aroua, 2007) have been investigated as adsorbents for the CO_2 separation and capture by selective adsorption. Among these materials, activated carbon is the most commonly used and most effective modified adsorbent support because of its high specific surface area and big pore volume. And the activated carbon by microwave heating processing, has the advantages of saving energy, space and time; reducing in the environmental impact of material processing and producing new materials and microstructures that cannot be achieved by other methods(Guo, et al., 2009),(Li, et al., 2009). Other than that, microwave radiation can generate hot spots (as a consequence of mineral impurities) inside the carbon particles where the temperature is much higher than the overall temperature of the sample. This temperature difference usually causes heterogeneous reactions between the sample and the inert gases that are taking part in the reaction. Additionally, it is nearly impossible to accurately measure the sample temperature, and only the surface temperature of the sample is measurable using an infrared pyrometer(Hesas, W. Daud, Sahu, & Arami-Niya, 2013). The internal temperature of the sample may be tens or hundreds of degrees higher than the sample surface temperature due to the internal and volumetric nature of microwave heating. Hence, the temperature could not be a variable condition in the preparation of AC using the microwave irradiation method. With the development of technology, the use of microwave-activated carbon received greater attention from researcher particularly because of the low producing cost, low energy requirement, and uncomplicated process and fine adsorption performance. The microwave-activated carbon was widely used for wastewater treatment(Huang, Sun, Wang, Yue, & Yang, 2011). However, investigations concerning the adsorption separation of CO_2 on microwave-activated carbon have not been much reported up to now.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

Before determining experimental methodology, extensive research was done to obtain an executable as well as feasible project plan. In order to get the key understanding on the effect of activation temperature and time on activated carbon from banana peel using conventional and microwave-heating method, journals, research papers, and articles were read and analyzed. To come up with suitable experimental procedures, the procedures of those researches were analyzed and compared. Thus, following the literature review, a project plan was developed in order to successfully achieve the project objectives:

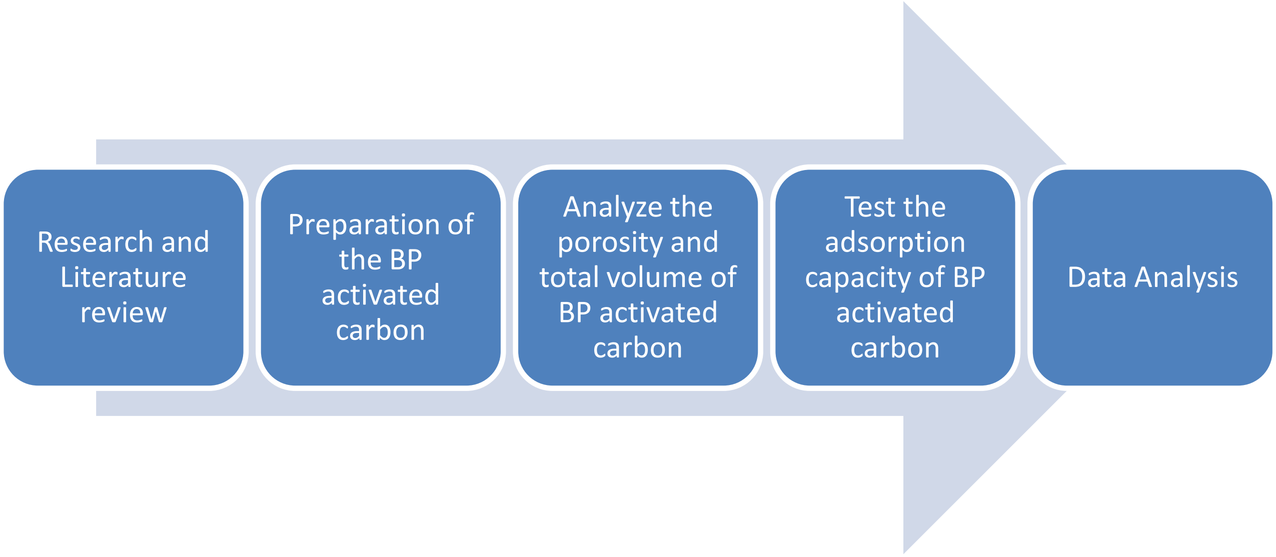

Figure 5 : Project Plan

3.2 Experiments Methodology

3.2.1 Banana Peel Sample Preparation

Banana peel (BP) is massively collected from fried banana stalls and cafeterias. After collecting sufficient BP, they are roughly cut into small pieces so that they can be thoroughly washed with tap water to remove external dirt. Then, the wet banana peel are kept in the oven for removing the water from the surface and dried at 100°C for minimum of 12 hours to remove excessive moisture. The dried banana peel are then grinded into fine powder form by using 0.25mm sieve when grinding, and then kept in an air tied container for further activation.

Figure 6: Fine banana peel powder

3.2.2 Chemical activation of Adsorbent

Chemical activation is used in this experiment as a variable to compare whether chemical activated carbon or physical activated carbon is the better adsorbent for carbon dioxide adsorption. Chemical activation is used in this study due to the lower temperature requirement and ability to gain higher yield with better porous structure in the activated carbon produced. The parameters which are manipulated in the activation phase of the BP based adsorbent are the types of activation temperature and activation time. The activating agents being used in this study are potassium hydroxide (KOH). This activating agents are chosen in this study based on the past researches conducted on similar agricultural waste precursor and the results of using these chemicals show better pore formation in the activated carbon compared to others. The range of activation temperature studied in this project are 400℃, 500℃ and 600℃. While the activation time used for chemical activation using conventional-heating method are two hours and three hours. The impregnation ratio used is constant as 1:1 (wt%). Impregnation ratio is defined as the dry weight of the banana peel powder, W_{BP} over the dry weight of activation agent used, W_{KOH}.

$$Impregnation\, ratio = \frac{W_{BP}}{W_{KOH}}$$

10 g of KOH is stirred is 100 mL of distilled water then 10 g of banana peel powder is mixed with the mixture. The mixture is left for an overnight to let the chemical mixed up well with the banana peel powder. The beaker containing the mixture of banana peel powder is covered with a piece of aluminium foil to avoid caontamination.

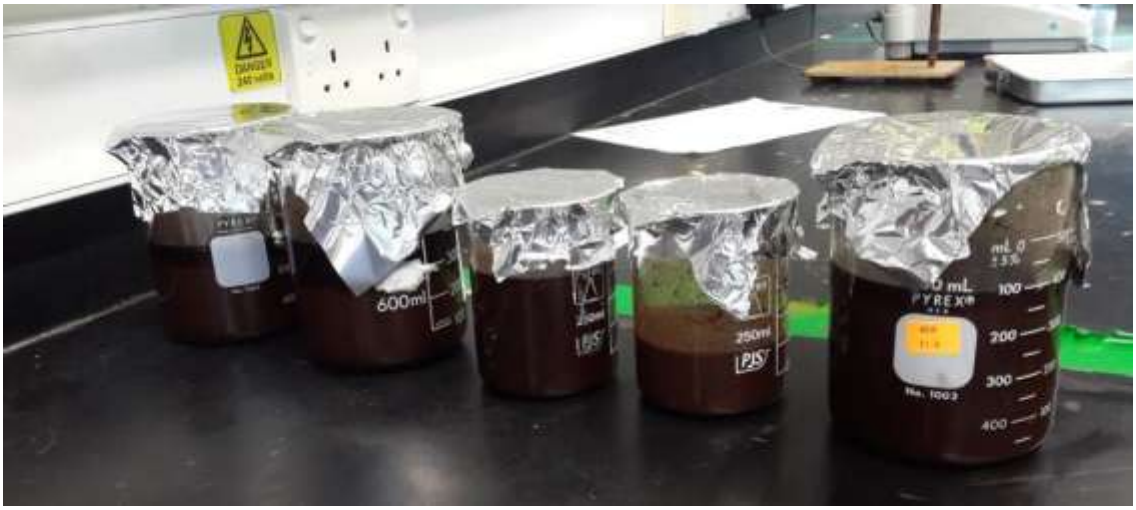

Figure 7: Banana peel powder soaked in KOH

Then, it will be filtered by using filter paper for another overnight and then dried in oven with 85℃ for 48 hours. Next is to activate the adsorbent, nitrogen gas will be used to purge the furnace at rate of $(100cm^3/min)$ and heating gradient at $10℃/min$ for the scheduled time. This is the point where the banana peel turns to activated carbon(Rashidi, Yusup, & Borhan, 2014). After being cooled off, all the samples are washed repeatedly until distillate turns to clear water. This is to ensure that there is no traces amounts of impurities that may block the pores of the activated carbon. Thus, it will disturb the process of adsorption in the test later on. The samples that had been carbonized for two hours will then be further heated by microwave-heating method with $560W$ for two minutes. Then, both sample, further heated and without further heating process activated carbon, will be send to the lab to analyze the porosity and total volume of pores. Then they will be tested for adsorption capacity. Several figures are shown in the appendices for the flow of preparing the activated carbon.

3.3 Characterization Of Sample

There are several equipments that are involve in characterizations of the samples. First and foremost, FESEM is used to provide magnification (10 – 10,000 times) to view the surface morphology of the activated carbon in high resolution images in order to confirm the formation of pores inside the activated carbon such as macropores, mesopores and micropores by comparing with the SEM images taken before the activation process. The characteristics generated by X-ray are used for identification of distinct elements present in the sample using energy dispersive X-ray spectrometer (EDX). The SEM's EDX feature is used to detect the element composition in the samples whether the carbon content in activated carbon of banana peel is enough for commercial production of activated carbon. By this step, the element composition in the sample could be defined by using the banana peel powder prior to activation and after activation.

Figure 8: Field Emission Scanning Electron Microscope (FESEM)

Moreover, the pore size distribution, specific surface area and as well as the porosity of the samples are determined by the nitrogen adsorption-desorption isotherms characterized by the Micromeritics ASAP 2020, by using nitrogen as the adsorbate while being degassed at 350℃ for four hours. The specific surface area of the activated carbon samples are estimated using the Brunauer-Emmett-Teller (BET) method using N_2 adsorption isotherm data while for the pore size distribution, the Barett-Joyner-Halenda (BJH) adsorption model is used(Borhan & Kamil, 2012). The formation of mesopores or smaller is more desired in this study to be qualified as activated carbon(Borhan & Kamil, 2012).

Figure 9: Micromeritics ASAP 2020

Lastly, in order to study the carbon dioxide adsorption capacity on activated carbon derived from banana peel, the High Pressure Volumetric Analyser (HPVA-II) was used. The HPVA Series of gas adsorption analysers from Particulate Systems is designed to obtain high-pressure adsorption isotherms using gases such as hydrogen, methane, and carbon dioxide using the static volumetric method. The volumetric technique consists of introducing (dosing) a known amount of gas, carbon dioxide in this case (adsorptive), into the chamber containing the sample to be analysed. When the sample (activated carbon) reaches equilibrium with the adsorbate gas, the final equilibrium pressure is recorded. These data are then used to calculate the quantity of gas adsorbed by the sample. This process is repeated at given pressure intervals until the maximum preselected pressure is reached. Each of the resulting equilibrium points (volume adsorbed and equilibrium pressure) is then plotted to provide an isotherm. Excellent reproducibility and accuracy are obtained by using separate transducers for dosing the sample and for monitoring the pressure in the sample chamber.

Figure 10: HPVA-II for Carbon Dioxide Adsorption Analysis

3.3.1 Tools and Equipment

Table 1: List of tools and equipments

No	Equipment	Uses
1	Oven	To dry the banana peel
2	Tubular furnace	To carbonize banana peel powder by conventional-heating method
3	A modified microwave oven	To further carbonize banana peel powder by microwave-heating method

No	Equipment	Uses
4	Field Emission Scanning Electron Microscope (FESEM)	To provide magnified view of the activated carbon structure derived from banana peel
5	Surface Area Analyzer and Porosimetry Systems (MICROMERITICS ASAP 2020)	To determine the pore size distribution, specific surface area and porosity of the banana peel activated carbon through nitrogen adsorption-desorption isotherms
6	HPVA II Analyzer	To test the adsorption capacity of carbon dioxide on prepared activated carbon

3.3.2 Substance and Chemicals

Table 2: Lists of substances and chemicals used

No	Materials	Purposes
1	Banana Peels	To be used as raw material to develop activated carbon in removal of carbon dioxide
2	Potassium Hydroxide (KOH)	To impregnate powdered banana peel by means of chemical activation to increase pore volume for adsorption

3.4 Gantt Chart and Key Milestones

No.	Detail / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic	■	■												
2	Preliminary Research Work		■	■	■	■	■								
3	Submission of Extended Proposal							■							
4	Proposal Defense								■	■					
5	Project work continues										■	■	■		
6	Submission of Interim Draft Report													■	
7	Submission of Interim Report														■

No.	Detail / Week	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29
1	Project Work Continue	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦		
2	Submission of Progress Report								🟦							
3	Project Work Continues								🟦	🟦	🟦	🟦	🟦			
4	Pre – SEDEX											🟦				
5	Submission of Final Draft Report													🟦		
6	Submission of Dissertation (soft bound)													🟦		
7	Submission of Technical Paper															🟦
8	Viva														🟦	
9	Submission of Project Dissertation (Hard Bound)															🟦

🟦 Process

CHAPTER 4

RESULTS AND DISCUSSION

4.1 FESEM Imaging and Elemental Composition Analysis

The production of activated carbon is defined as the fraction of dry weight of activated carbon yielded to that of the powdered banana peel. The formula for the calculation of yield (Diao, Walawender, & Fan, 2002) is as below:

$$Yield = \frac{M_{AC}}{M_{BP}} \times 100\%$$

Whereby:

M_{AC} = dry mass of the activated carbon after washing (g)

M_{BP} = original mass of the banana peel powder before carbonization (g)

Table 3: Elemental Composition for RAW Sample and Sample B2

Element	Before Carbonization (RAW)		After Carbonization (B2)	
	Weight %	Atomic %	Weight %	Atomic %
Carbon, C	58.67	65.64	56.18	70.55
Oxygen, O	25.78	27.39	27.19	25.57
Silicone, Si	0.38	0.21	0.68	0.34
Potassium, K	15.17	6.76	15.95	3.54
Total	100	100	100	100

The converted activated carbon reached an average yield of **45.67%**. It is confirmed that the weight of banana peel for all the samples is fixed at 10g but there are some loss in banana peel powder mass during the transfer from the filter paper to the crucible for carbonization, during the washing and other stages along the point to reach the characterization machines. Therefore, the tabulated mass of banana peel powder in APPENDIX shows less than 10g.

It is determined by Energy Dispersive X-ray (EDX) Spectroscopy that the carbon content (weight) of raw banana peel sample is **58.67%** which indicates that banana peel is appropriate to be used as activated carbon since its carbon content falls within the desirable range (50% – 80%)(Wu, Tseng, & Hu, 2005).

The oxygen, O content is also higher in sample B2. These observations can be caused due to the insufficient washing of the samples after the overnight impregnation with chemical reagent. Silicon can be considered as impurities in the sample which possibly originated from the crucible that holds the sample in the furnace during the carbonization process. There are traces amount of potassium which is consider normal, because potassium is a common element found in banana fruit. On the other hand, sample B2 has been impregnated with potassium hydroxide (KOH) solution and carbonized. This is portrayed in the EDX analysis for the sample, as potassium, K content is found among other elements.

The following are the results of FESEM imaging done. The sample of A1, A2 and A3 are taken from another research (Ramdan, 2014). Based on the FESEM imaging, Figure 11 displays the passage construction in raw banana peel sample, which is essential in production of activated carbon. The canal permits the banana peel powder to absorb the chemical activation agent to activate pore development. On the other hand, Figure 12 - 18 is the 1000 times magnification on all of the samples. The image clearly shows that sample A1 already have pores formation at 400°C and two hours of carbonization. More well-structured pores are observed on the surface morphology of sample A2, A3, B1, MW1, MW2 and MW3 as shown in Figure 13-18. From Figure 15 of sample B1, at 400°C of carbonization for three hours shows that the pores are getting bigger after a longer time of carbonization. At 500°C of carbonization temperature for two hour for sample A2 from Figure 13, the porous wall are broken slightly but based on Figure 17 from sample MW2 when heated with microwave after carbonization, the porous wall are further enhance with more pores. However, Figure 14 shows the effect of implying excessive heat during carbonization, which is 600°C for two hours and by further heating it with microwave in Figure 18. This indicates that at 600°C of temperature, the heat is excessive for the sample which results in the knocking and breaking of the porous formation in the sample (Borhan & Kamil, 2012). In addition, the carbonization period is also too long which deteriorates the formation of activated carbon.

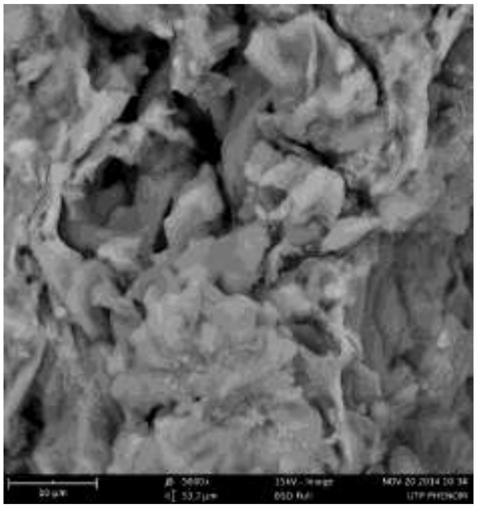

Figure 11: RAW Sample SEM image

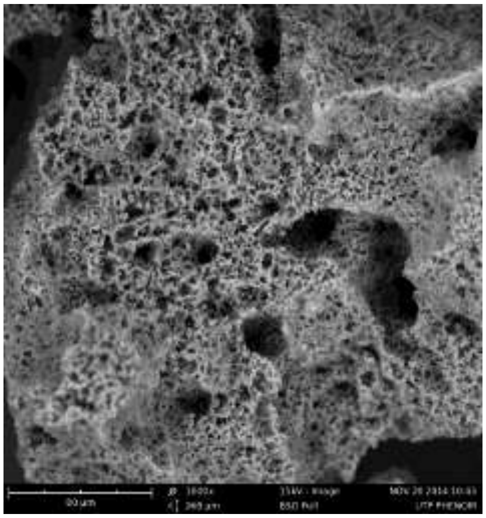

Figure 12: Sample A1 SEM image

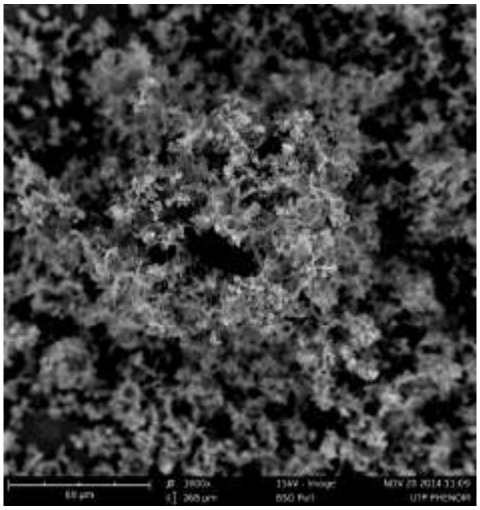

Figure 13: Sample A2 SEM image

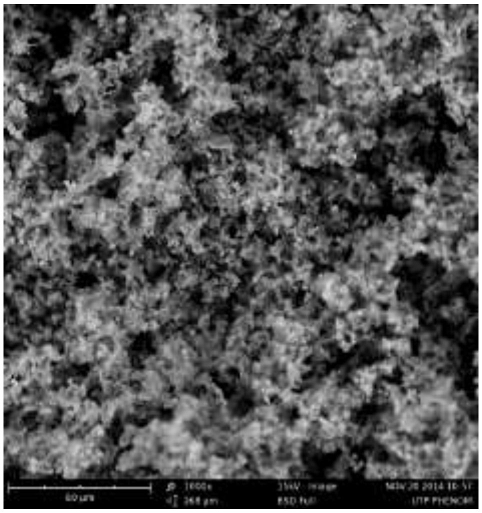

Figure 14: Sample A3 SEM image

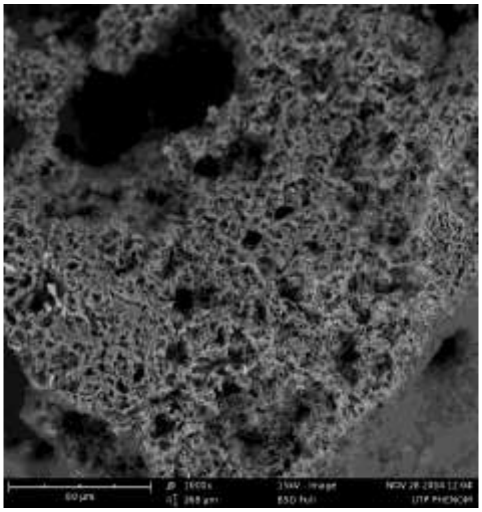

Figure 15: Sample B1 SEM image

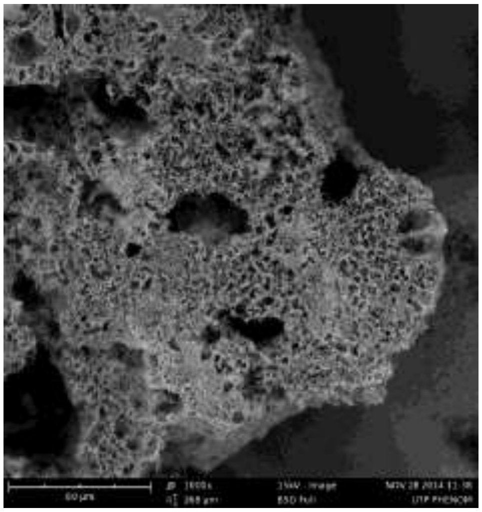

Figure 16: Sample MW1 SEM image

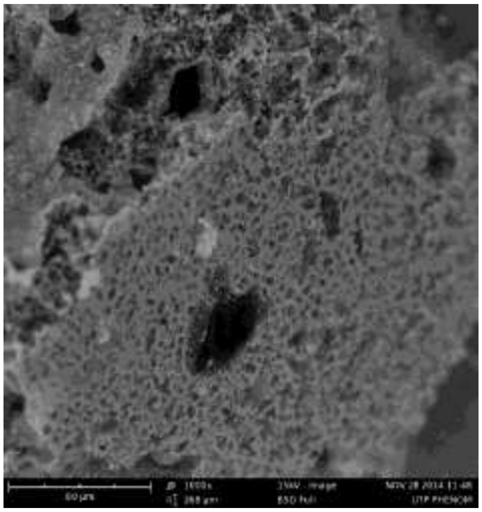

Figure 17: Sample MW2 SEM image

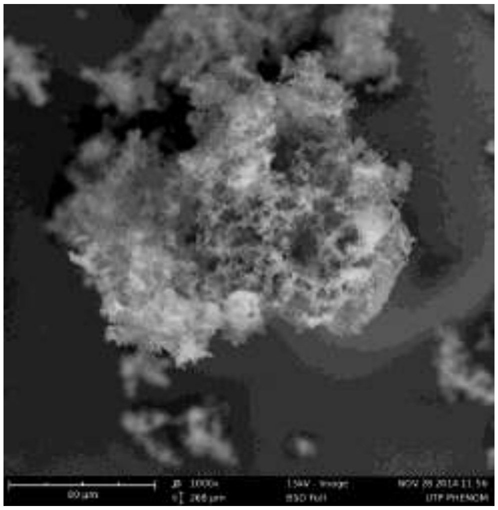

Figure 18: Sample MW3 SEM image

4.2 Surface Area and Porosity Analysis

Table 4: Surface area and porosity results for selected samples(Thangamuthu, 2014)

Sample Label	Activation Temperature (^oC)	Activation time (hours)	Heated by microwave	S_{BET} (m^2/g)	V_T (cm^3/g)	D (nm)
A1	400	1	No	31.4678	0.01593	4.5137
A10	400	2	No	260.3841	0.01436	0.2508
B1	400	3	No	11.5140	0.014207	4.93567
B2	500	3	No	341.7777	0.215119	2.51764
B3	600	3	No	501.3886	0.301132	2.40238
MW1	400	2	Yes	8.5713	0.026022	12.14375
MW2	500	2	Yes	1142.7893	0.636435	2.22765
MW3	600	2	Yes	889.9308	0.485196	2.18149

The table above shows the resulting surface area of the pore formed on activated carbon derived from banana peel at different preparation conditions. Result A1 and A10 are taken from another research (Thangamuthu, 2014). Based on the results, it shows that 500°C is the maximum carbonization temperature for banana peel based activated carbon.

Samples A1, B1, and MW1 are all carbonized at 400°C each with the activation time of two hours, three hours and two hours respectively, yield low S_{BET} . The other samples, B2, B3, MW2, and MW3 which is carbonized more than 400°C for their respective activation time base on the Table 4 rather yield quite high S_{BET}. In addition, group B samples which are carbonized for three hours yield larger surface area than sample A1 and A10 which are carbonized for two hours but to the exception with sample B1 where it just yield very low surface area comparing to A1. This may happened due to the exposing the activated carbon for a period of time outside the air tight container. Other than that, sample group MW which is carbonized for two hours with respective activation temperature from Table 4 then further heated with microwave yield larger S_{BET} compared to sample group A which are only carbonized without further heating with microwave yield less S_{BET}. Moreover, all of the sample are grinded to 0.25mm in size which is comparatively should yield larger surface area according to phenomenon occurs because larger particle size exposes less surface area for activation during impregnation phase, thus resulting in smaller S_{BET} (Borhan & Kamil, 2012).

Commonly, pore broadening will occur due to the rise in activation temperature and activation time. Due to carbon gasification enhancement with sufficient KOH, the pore size in activated carbon is enlarged together with the porosity. Raising the activation temperature directly increases the reaction rate of C-KOH reaction, causing increased carbon burn-off. Since KOH reagent is a strong base, it enables the interface with carbon atoms to increase the rate of dehydrogenation and oxidation, inviting the rise in tar formation and development of porosity (Mopoung, 2008).

Lastly, it is also been proved that all the samples analysed are at most to contain pores of class mesoporosity. The results also shows that sample MW2 yields the biggest surface area, S_{BET} with area 1142.7893 m^2/g with pore diameter 2.22765 nm of mesoporous group. But, since sample B2 exhibit the best result among the first batch for surface area and porosity analysis which consist of sample B1, B2 and MW1, thus this sample was utilized to study the adsorption capacity of carbon dioxide.

4.3 Nitrogen Adsorption-Desorption Isotherm

Figure 19 shows the nitrogen adsorption-desorption analysis for selected samples. This study is done to identify the correct adsorption isotherm type based on the IUPAC classifications as shown in APPENDIX. In order to satisfy the adsorption analysis, the adsorption isotherm should at least fit one or a combination of the six distinct isotherm graphs (Fletcher, 2008). As shown in Figure 19, all the selected samples are identified to follow the isotherm similar to TYPE II and TYPE III. TYPE II isotherm specifies that an unlimited layer formation after completing the monolayer observed in adsorbents with a varied pore dimensions. Besides that, this type of isotherm typically refers to poly-molecular adsorption in either nonporous or macro-porous adsorbents. However, this type of isotherm do not exhibit saturation limit. In addition, TYPE III isotherm usually achieved with respect to increasing amount of adsorbed gas without constraint as its relative saturation achieves unity. TYPE III isotherm also displays characteristics of non-porous sorbents with low energy of adsorbent-adsorbate interaction. Therefore, these two types of isotherm are closely related to the gas-solid adsorption of carbon based material with mesoporosity to macroporosity. They also have a mixture of strong and weak adsorbate-adsorbent interaction. Since these samples proved larger surface area with mesoporosity range of pore diameter, hence their isotherms tend to follow TYPE II and TYPE III isotherms which described the isotherm of larger porosity. Figure 19 also illustrates the adsorption isotherm for the largest surface area yielding sample, that is, sample MW2. Adsorption isotherm of MW2 clearly follows TYPE III isotherm which is related to weak interaction of adsorbent-adsorbate, commonly found in physical adsorption of gases of microporous adsorbent. This result is evident with BET surface area analysis proving that the pores are in mesoporosity group with pore diameter, 2.22765 nm.

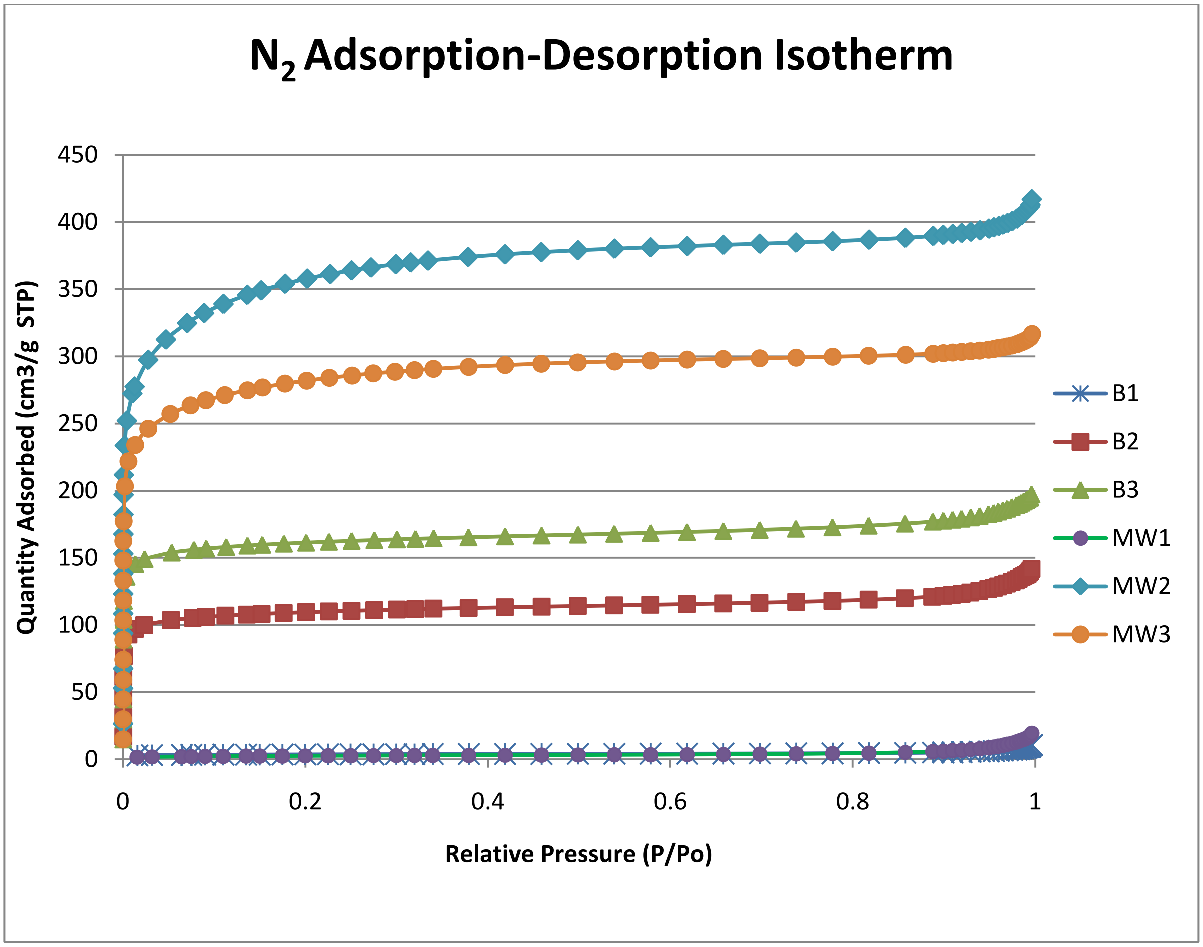

Figure 19: Nitrogen Adsorption-Desorption Isotherm for selected samples

4.4 Carbon Dioxide Adsorption Analysis

Figure 20 shows the amount of carbon dioxide adsorbed in weight percentage with increasing pressure. The adsorption test is carried out using HPVA II analyzer at temperature of 25°C. Both sample MW1 and B2 are able to show that the amount of carbon dioxide adsorbed increases with increasing pressure, proving that activated carbon derived from banana peel is capable in removing carbon dioxide gas with mesoporous pore formation. The difference in the line graph is due to the adsorption capacity by the samples itself, the sample B2 have a higher S_{BET} (341.7777 m^2/g) and sample MW1 has a lower adsorption capacity by having lower S_{BET} (8.5713 m^2/g).

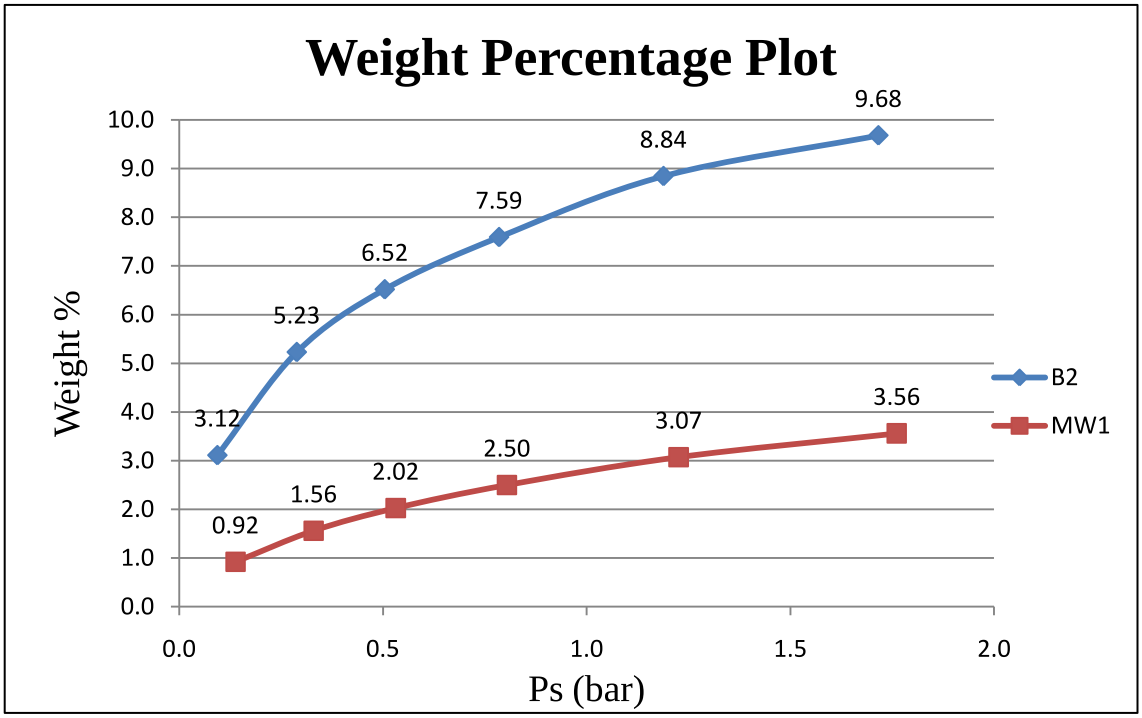

Figure 20: Weight percentage of carbon dioxide adsorbed

The adsorption isotherm in Figure 21 seemingly have the identical shape to the isotherm in Figure 20 which is the Type I isotherm. The adsorption isotherm shows decreasing tendency with increasing temperature. Thus, carbon dioxide adsorption is obviously excellent at lower temperature because it is an exothermic process, whereby heat is released (Esteves, Lopes, Nunes, & Mota, September 2008).

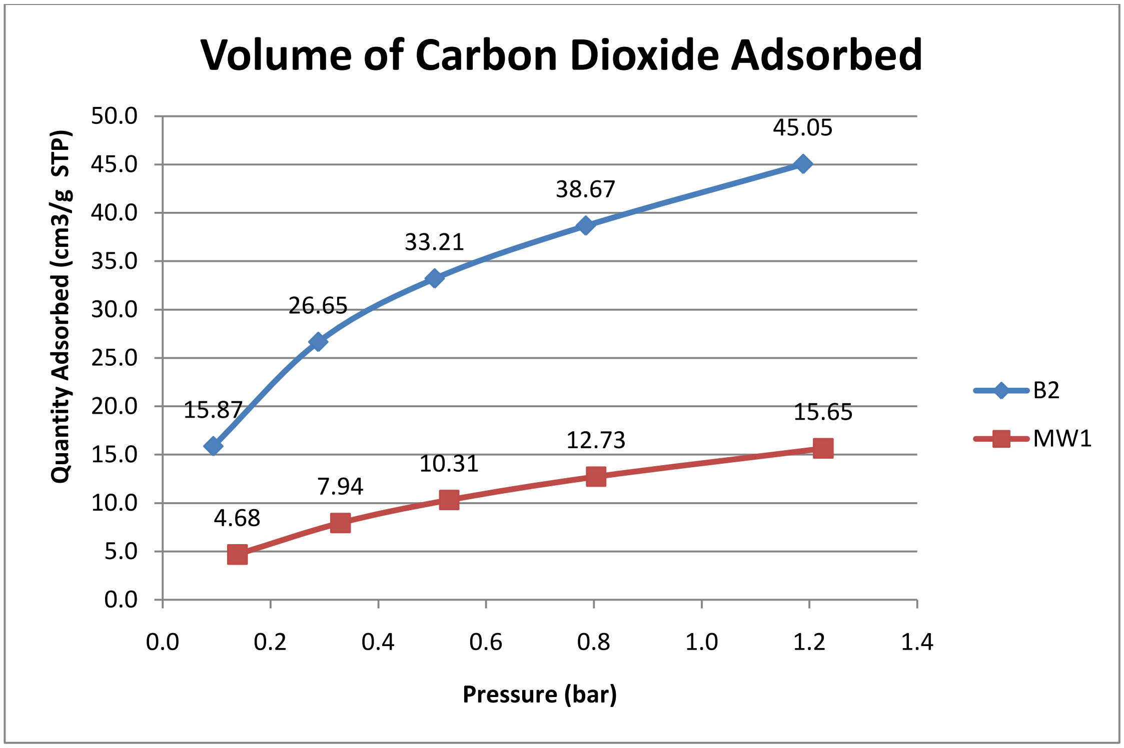

Figure 21: Volume of carbon dioxide adsorbed

Isotherm Type I implies the features of reserved to adsorption by a few molecular layer. The pores do not leave empty spots for further adsorption once the adsorbates have occupied it. Besides that, Type I isotherm is also known as pseudo-Langmuir isotherm as it shows monolayer adsorption well. Type I is found in chemisorptions where the converging approach to limiting the quantity that all of the surface of pores are occupied. While for physisorptions, the Type I isotherm are found with mircoporous powder that are having pore size that does not overtake the adsorbate molecular diameters. In these small dimension, gas molecule are found to overlap to the pore walls which increase the quantity of gas adsorbed at relatively low pressures. As the pressure increases, the adsorbed adsorbates have filled the pores and condensed to point into an almost static adsorption showing very little of none adsorption. Physisorption that produces the Type 1 isotherm shows that exposed surface are categorized within the micropores, which once have adsorbed adsorbate, will leave little or no surface for further adsorption.(Heidari, Younesi, Rashidi, & Ghoreyshi, March 2014)

Comparatively, the adsorption isotherm shown by other identical research on developing activated carbon from various precursors for the purpose of removing carbon dioxide is shown in figures below. It is also proven that the entire adsorption isotherm follows the Type I isotherm which is identical to Figure 21. By comparison, the carbon dioxide uptake on activated carbon developed from banana peel seems slightly higher. Hence, banana peel based activated carbon shows better adsorption capability compared to other activated carbon from different precursors like petroleum pitch and carbon by 5%.

The effect of temperature on the amount of carbon dioxide, CO_2 adsorbed onto the activated carbon prepared with potassium hydroxide, KOH, decreases with increasing temperatures as shown in Figure 22. In other words, the result clearly illustrates that CO_2 adsorption was most significant at lower temperature with bigger adsorption capacity proving that physical adsorption has taken place (Heidari, Younesi, Rashidi, & Ghoreyshi, March 2014). Therefore, it is reasonable that adsorption test for carbon dioxide on banana peel based activated carbon is studied at low temperature of 25°C.

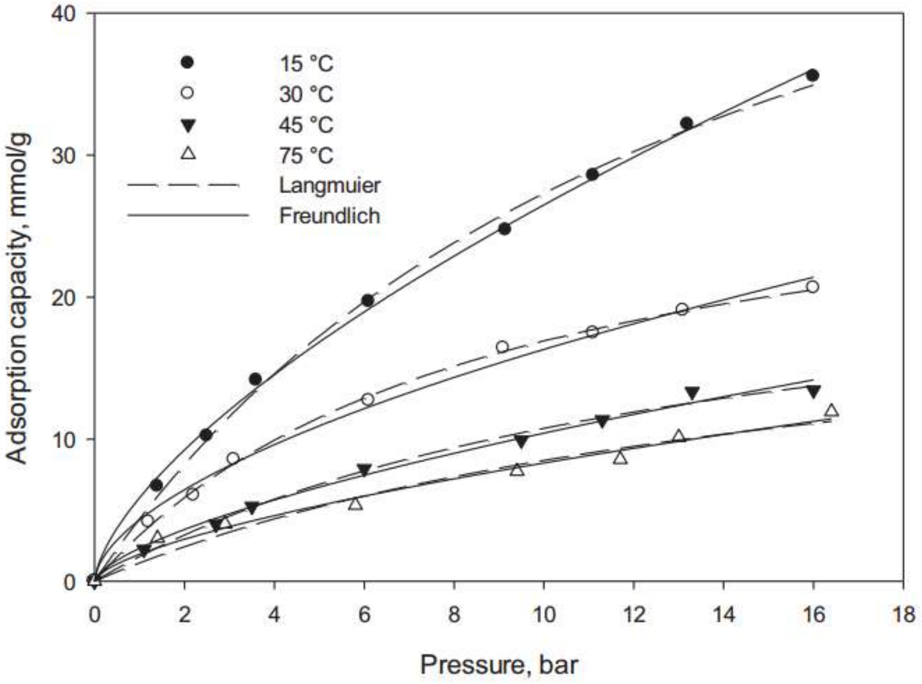

Figure 22: Adsorption capacity of carbon dioxide on activated carbon derived from *Eucalyptus camaldulensis* wood at different temperatures (Heidari, Younesi, Rashidi, & Ghoreyshi, March 2014)

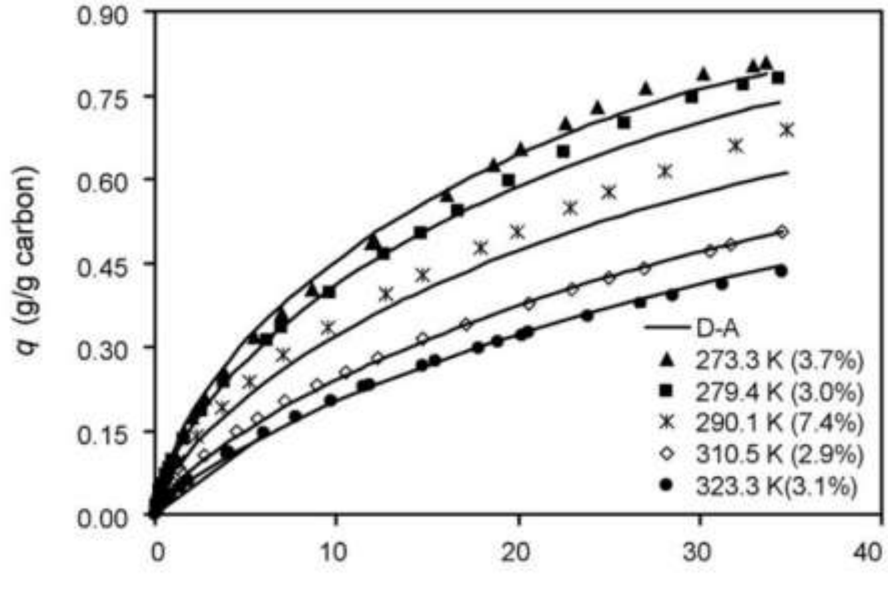

Figure 23: Adsorption isotherm of activated carbon derived from coal, extruded carbon (Esteves, Lopes, Nunes, & Mota, September 2008)

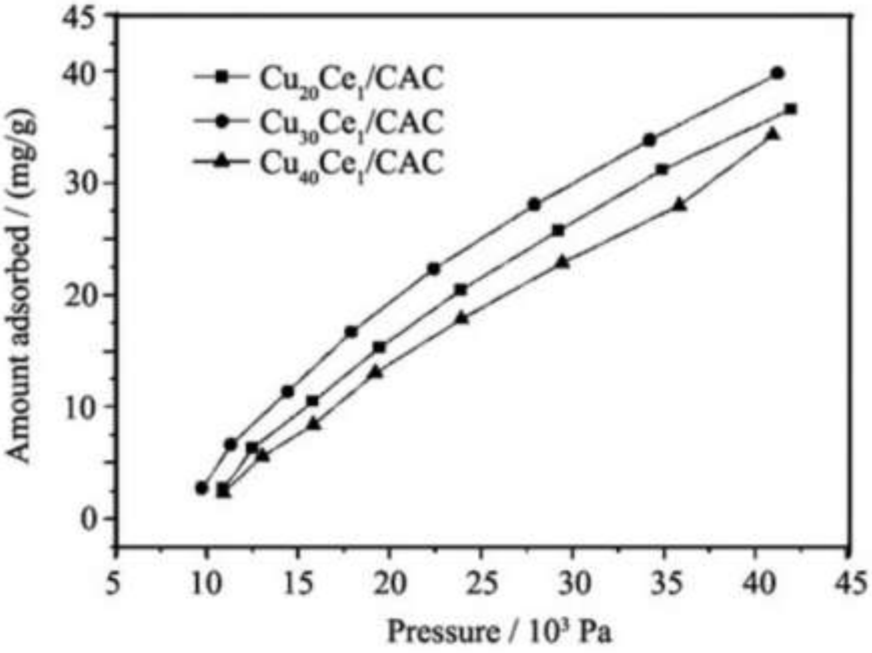

Figure 24: Adsorption isotherm of activated carbon derived from coconut (Fenrong, Honghong, Xiaolong, Ping, Qiongfen, & Dongjuan, December 2010)

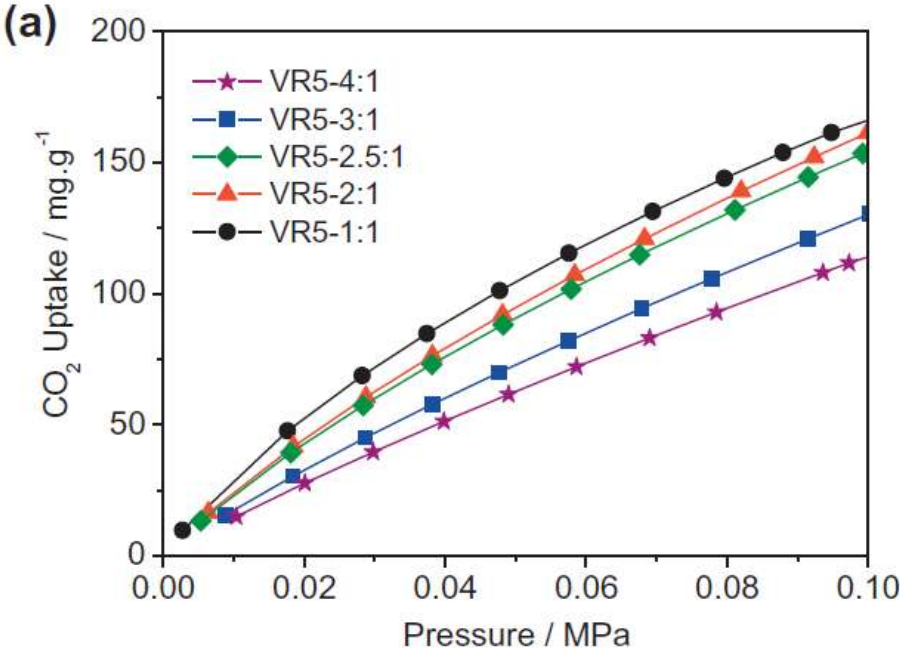

Figure 25: Adsorption isotherm of activated carbon derived from petroleum pitch (Casco, Martinez-Escandell, Silvestre-Albero, & Rodriguez-Reinoso, February 2014)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.2 Conclusion

As a conclusion, this project is important as it deals with alternative ways of removing carbon dioxide emissions in atmosphere. Adsorption using activated carbon should be further studied to prove that it can be one of the effective ways to deal with the current issue of using banana peel in conventional ways especially in eliminating carbon dioxide. Thus, the investigation in this project shows that the banana peel is an appropriate precursor to be adapted into activated carbon due to its high carbon content. Chemical activation method is preferred in this study with the usage of common chemical activation agents such as potassium hydroxide. In conjunction with that, various preparation parameters have been studied thoroughly to study the effects on surface area and pore formation of the derived activated carbon from banana peel. Therefore, referring to the results, it is proven that smaller particle size is better to be utilized as chemically activated carbon as it exposes larger surface area during impregnation and activation. In addition, the impregnation ratio should be fairly low to inhibit good pore formation. Furthermore, the carbonization temperature for using banana peel as precursor should not be higher than 500°C as any higher will bring about the breakdown of the porous formation in the activated carbon and cause the reduction of effective surface area for the adsorption process. Sample B2 proved to be the best sample among others with its outstanding BET surface area as well as nitrogen adsorption-desorption isotherm indicating mesoporosity. Lastly, activated carbon derived from banana peel also proved to remove carbon dioxide through adsorption.

5.2 Recommendation

The project can be improved to the next level of research, that is, to study the adsorption as well as desorption process using the banana peel based activated carbon as adsorbent. In terms of adsorption, there are ample of parameters that can be considered such as the amount of the adsorbent present, the pH of the solution, the temperature, the optimum condition for microwave heating and contact period of the adsorption process. The sample with the highest S_{BET} from this project (sample MW2) will be produced in bulk quantity to conduct this study. The study on the adsorption process is significant in determining the most conducive environment for the greenhouse gas to be adsorbed on the reactive sites of the banana peel based activated carbon which will highly increase the adsorption capacity of the adsorbent. The time desired to reach the adsorption equilibrium can be determined through this study. Adsorption equilibrium is achieved when the rate of adsorption is equal to the rate of desorption, thus making any further adsorption highly unlikely (maximum amount of adsorbate bind on the reactive sites of the adsorbent). On the other hand, study on desorption process also will be helpful in discovering conservational way of removing harmful gases such carbon dioxide. This research would be significant in the effort to reduce air pollution as well as eliminating greenhouse gases in a safe manner.

In addition, different types of biomass or agriculture wastes can also be examined on the feasibility as precursor to be converted into activated carbon. Activated carbon made from different precursor will have different preference or adsorption capacity, depending on its respective preparation conditions. Additional information can be obtained through similar research with different raw materials in order to identify better bio-sorbent to remove pollutants. Hence, the study of developing adsorbent derived from agricultural waste would be a great exploration to deal with as it gives a better alternative economically as well as environmentally to preserve pollution-free environment.

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Appendices

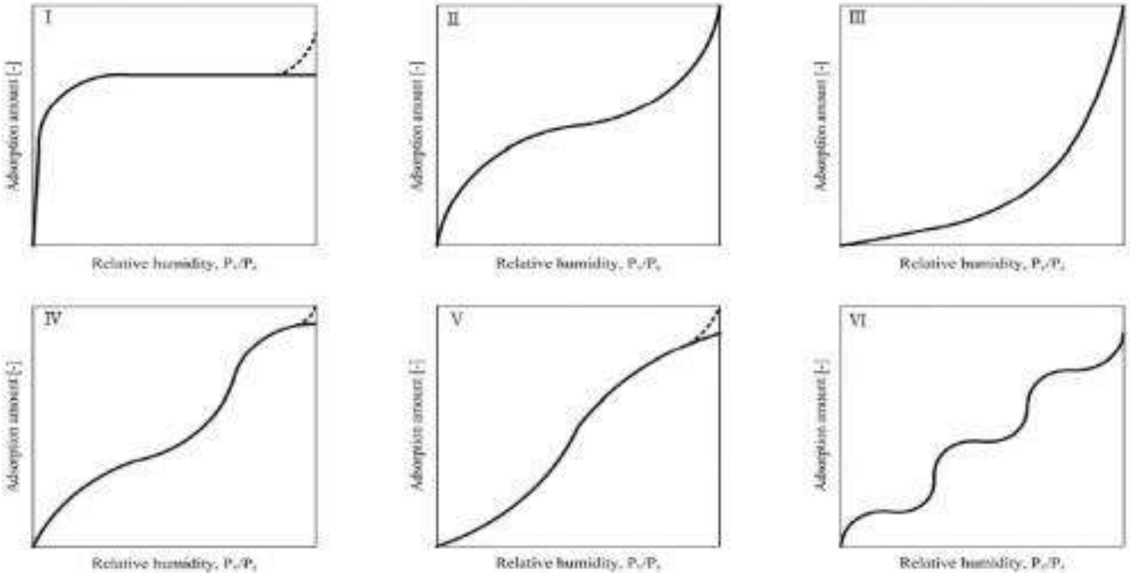

Figure 26: BET Adsorption Isotherms (Kim & Chea, 2012)

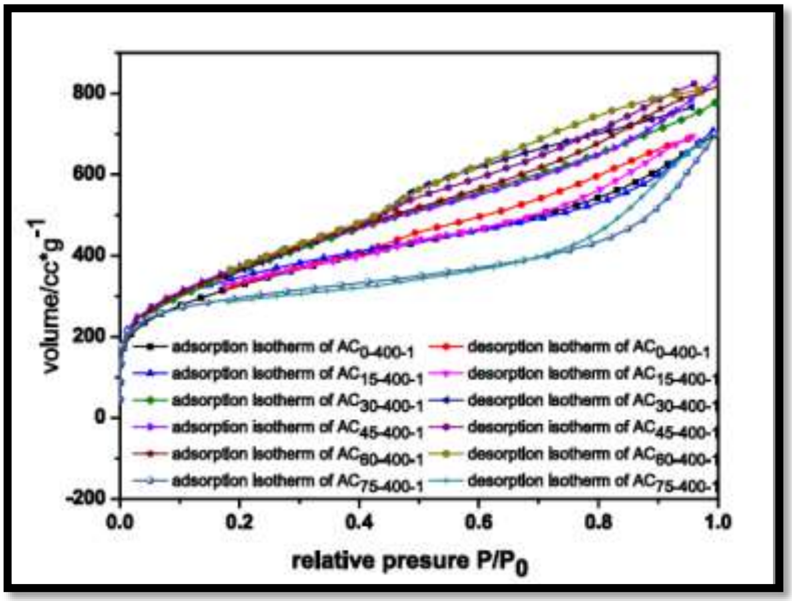

Figure 27: Adsorption-desorption isotherm of N_2

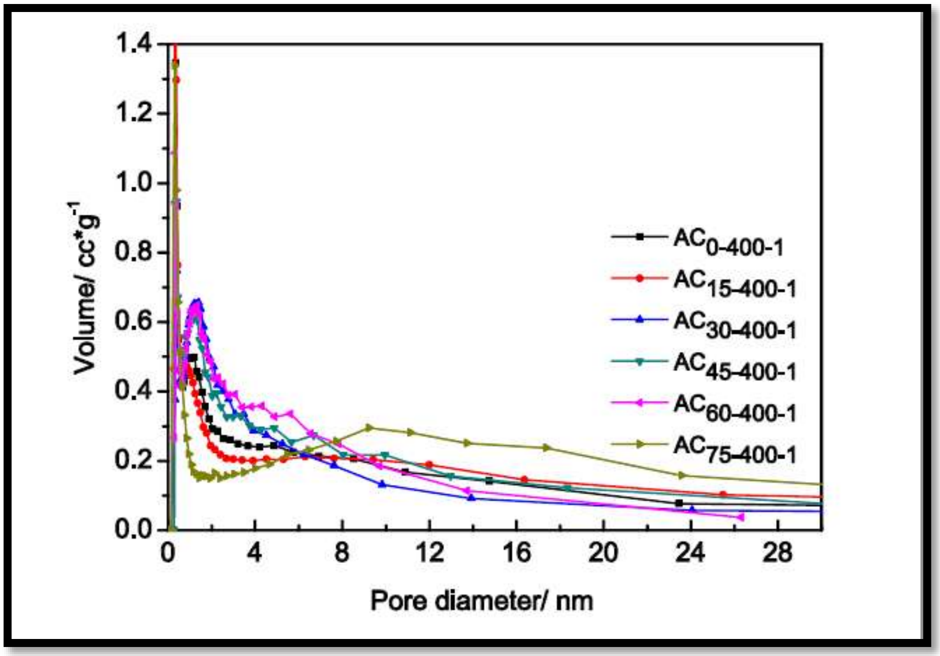

Figure 28: Pore size distribution of activated carbon

Figure 29: Raw banana peel

Figure 30: Dried banana peel

Figure 31: Filtered impregnated banana peel powder

Figure 32: Dried impregnated banana peel powder

Figure 33: Activated carbon from banana peel