Development of Activated Carbon Derived from Banana Peel for Carbon Dioxide Removal

by

Subhashini Thangamuthu

15155

Dissertation submitted in partial fulfilment of the requirements for the

Bachelor of Chemical Engineering (Hons)

May 2014

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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In partial fulfilment of the requirement for the

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Approved by,

(Mr.Azry Borhan)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 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.

SUBHASHINI THANGAMUTHU

ABSTRACT

The focal point of this research is to study the prospective of using banana peel as potential adsorbent for eliminating carbon dioxide from polluted air in the atmosphere. This project work highlights on the constraints involved in the preparation of the banana peel bio-sorbent, such as activating agent, impregnation ratio, activation temperature and period of activation. Initially, the withered banana peel is grinded into powder form, then chemically activated using potassium hydroxide and activated at altered ranges of temperature and duration. This carbonization process allows the formation of activated carbon, one of a common type of adsorbent used globally especially in industries' effluent treating. Micromeritics ASAP 2020 and Field Emission Scanning Electron Microscope were used in identifying the best sample preparation method with the largest surface area which directly contributes to the effectiveness of adsorbent in removing carbon dioxide (CO₂). In addition, the activated carbons developed from banana peel are also verified for their adsorption capacity with harmful greenhouse gas such as carbon dioxide using HPVA II analyzer. The samples were also analysed for its elemental composition before and after carbonization which results in 60.87 wt% and 58.18 wt% of carbon content respectively. Sample A10 was identified to yield activated carbon with largest surface area, S_{BET} of 260.3941 m²/g and pore diameter, 0.2508 nm which falls in sub-microporous category. Sample A10 with particle size 0.25 mm was prepared using potassium hydroxide as activating agent with impregnation ratio of 1:1 and carbonized at 400°C for two hours. This sample of A10 is then further tested for the carbon dioxide adsorption test at 25°C which proved to adsorb carbon dioxide gas.

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CHAPTER 1

INTRODUCTION

1.1 Background

This project is related to the removal of carbon dioxide from gas/air streams focusing in adsorption by activated carbon extracted from banana peel wastes. Globally, fossil fuel is supplied worldwide with more than 98% of the energy necessities. Nevertheless, the burning of fossil fuels is one of the major contributors to the greenhouse gas, carbon dioxide, CO_2 . It is essential to develop technologies that will diminish greenhouse gas emissions in line with utilization of fossil fuel. Unfortunately, the current technology to eliminate or reduce the carbon dioxide concentration in air is costly and energy intensive. Therefore, enriched technologies for CO₂ capture are obligatory to achieve low energy drawbacks. Hence, adsorption has been identified as one of the potential methods for removal of CO_2 from gas streams. There are several ways in removing carbon dioxide such as chemical separation, membrane separation, and cryogenic separation as well as by adsorption. There has been plenty of research done on viability of transforming cultivated wastes into useful product such as 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 incorporates the effectiveness of the activated carbon in removing carbon dioxide. Remarkable adsorption effectiveness can be observed from activation of raw materials with porous structure. 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 the pore characteristics of activated carbon, adsorption isotherm and carbon dioxide adsorption capacity. Generally, banana plants are part of the Musaceae family whereas the scientific name for banana fruit is $Musa \times paradisiaca$. The primary purpose of the plant is to produce healthy fruits besides contributing to the manufacturing of fibre and being ornamental plants. The physical characteristics of banana plants are usually tall and sturdy. The stem of banana plant is capable of producing a bunch of green banana fruits which turn yellow in colour when mature. Commonly, banana fruits nurture in hanging bunches, with approximately 20 fruits to a hand and 3–20 hands to a cluster. One fruit of banana weighs 125g in average with 25% dry mass and the remaining 75% is water. Banana has been identified as the most common fruit with largest consumption worldwide, whereby the useless peels directly contributes to major agricultural waste. In order to overcome this environmental issue, banana peels have been tested and verified as effective adsorbents especially for heavy metals from industrial wastewaters as well as gaseous pollutants. Hence, it is significant to explore agricultural by-products that could be transformed to essential and resourceful products such as adsorbents.

1.2 Problem Statement

Since the 18th century, the industrial revolution began and there has been a rapid development in trades which directly contribute to the worldwide economy inclination as well as human development. Discharging of the pollutants is becoming the most provoking hazard to environment safety and even human beings' lives.

In this era of globalisation, as we are busy chasing after economic development, we neglect our mother nature and ignore its consequence despite instilling monetary values. As the individual living cost grows, we can see more cars and buildings all around but unfortunately less trees and plants. This definitely contributes to the greenhouse gases such as carbon dioxide which slowly depletes the ozone layer. Carbon dioxide is a gas primarily released from combustion processes and industrial plants, is the main contributor to global warming.

In recent years, the effects of greenhouse gases, especially carbon dioxide were often discussed as a vital environmental issue. Carbon dioxide is an essential and destructive greenhouse gas that is produced from increased burning of hydrocarbons due to the industrial rebellion contributing to global warming. It was reported that, since 1751 approximately 337 billion metric tonnes of carbon have been released to the atmosphere due to the fossil fuels ingestion and cement production (Boden, Marland & Andres, 2010). According to statistical survey, 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.

Global climate change will affect people and the environment in many ways. Some of these impacts, like stronger hurricanes and severe heat waves, could be life threatening. Others, like spreading weeds, will be less serious. However, as the Earth keeps getting warmer, the negative effects are expected to outweigh the positive ones. Humans and other living bio-organisms may face serious consequences due to global warming which include malaria, mental illness, loss of habitat, loss of healthy crops for food consumptions and more. Extreme heat can cause illnesses such as heat cramps, heat stroke, and even death. Certain kinds of air pollutants, like ozone, can make asthma and other lung conditions worse.



In conjunction with developing eco-friendly technology to reduce the carbon dioxide emissions, many researches have been done with different experimental methodology. One of the most economically and environmentally preferred technology is adsorption by agriculture wastes. Activated carbon was significantly plays a great role of adsorbent due to its large surface area and porosity which benefits it to be widely used for carbon dioxide removal (Jang & Park, 2014).

1.3 Objective

The key objective of this project is to assess the prospective of using banana peel as a potential adsorbent in removing carbon dioxide from the industrial effluent. Apart from that, the relationship between different preparation condition of the banana peel and the resulting adsorption capabilities of the adsorbent is studied to determine the pre-eminent preparation condition of converting banana peel to activated carbon. The adsorption capacity of activated carbon as an adsorbent, derived from banana peel in the removal carbon dioxide could also be examined from this project.

In short, the objectives of this project are:-

- 1. Preparation of activated carbon derived from banana peel by chemical activation
- 2. Characterization of pores of activated carbon by studying their pore size and adsorption isotherms accordingly
- 3. Utilization of banana peel for carbon dioxide adsorption

1.4 Scope of Study

This project emphases on defining the optimum preparation condition to derive activated carbon from banana peel which is used as adsorbent in removing carbon dioxide in the air. Biosorbent adsorption is believed to carry a significant value in removing pollutants or rather greenhouse gases such as carbon dioxide from the air. Therefore, activated carbon derived from agricultural waste such as banana peel is believed to perform as an efficient adsorbent to eliminate carbon dioxide. Hence, this project is relevant as development of activated carbon derived from banana peel for carbon dioxide removal has not been widely addressed yet. The experiment will use potassium hydroxide (KOH) as activation agent to activate carbon content in banana peel. 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. Impregnation ratio
- 2. Activation temperature
- 3. Activation time

The scope of the activation temperature is limited between 400°C to 600°C and the activation time is limited to 1 hour and 2 hours respectively. The impregnation ratio and the particle size are also among the parameters that are deliberate in order to determine adsorption effectiveness of banana peel based adsorbent. On the other hand, this project also discovers the effectiveness of the banana peel based adsorbent in terms of removing harmful greenhouse gas, that is, carbon dioxide.

1.5 Relevancy of Project

This project emphases on defining the optimum preparation condition to derive activated carbon from banana peel which is used as adsorbent in removing carbon dioxide in the air. Biosorbent adsorption is believed to carry a significant value in removing pollutants or rather greenhouse gases such as carbon dioxide from the air. Therefore, agricultural waste, activated carbon derived from banana peel is believed to perform as an efficient adsorbent to eliminate carbon dioxide. Hence, this project is relevant as development of activated carbon derived from banana peel for carbon dioxide removal has not been widely addressed yet.

1.6 Feasibility of Project

This project is feasible as it deals with narrowed scope of experiment whereby only three parameters are tested. It is within capability to be executed with helps and guidance from the supervisor and the coordinator. It is positive that this project can be completed within the time allocated with the acquiring of equipment and materials needed. The time frame for this project work is about 28 weeks.

CHAPTER 2

LITERATURE REVIEW

2.1 Activated Carbon

Activated carbon is known as charcoal that was preserved with oxygen to expose millions of tiny pores between the carbon atoms. Activated carbon possesses remarkable adsorptive capacity is vital for numerous dissolved organics and chlorine as well as the capability to adapt for precise application. Thermal decomposition method is used to convert carbon-based materials into activated carbon using furnace at controlled temperature and pressure. Adsorption takes place on the resulting huge surface with web of sub-microscopic pores. Amazingly, one pound of carbon provides a surface area equivalent to six football fields (Jang & Park, 2014).

2.2 Adsorption

Adhesion of particles of any form of matter to a surface is known as adsorption. A film of adsorbate on the surface of the adsorbent is formed due to adsorption. Comparatively, absorption includes the entire volume matter while adsorption takes place at surface of the material. Similar to surface tension, adsorption is a consequence of surface energy whereby, atoms fill in the bonding requirement of bulk material atoms. Nevertheless, atoms on the surface of the adsorbent are not completely enclosed by other adsorbent atoms and hence can invite adsorbates. Generally, the adsorption process is classified as physisorption or chemisorption. Activated carbon works by means of physical adsorption in removing water contaminants. Simply, physical adsorption occurs due to exertion of attractive forces by all molecules, especially molecules at the surface of a solid (pore walls of carbon). Eventually, these surface molecules hunt for other molecules to adhere to. Adsorption of water contaminants is due to the stronger attraction of the carbon surface compared to the attractive forces in solution. Neutral or non-polar organic compounds with lower water solubility and higher molecular weight are more adsorbable onto activated carbon.



Figure 6: Adsorption of contaminated particles

2.3 Banana Peel

A banana peel is the outer skin layer which covers the soft yellowish banana fruit. Bananas gained its popularity globally with yearly making approximately 145 million tonnes, whereby, with regard to its consumption, there is a contribution to enormous amount of banana peel waste 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. 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. 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 & Mazzoni, 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 & Mazzoni, 2014). Besides that, elements such as extractives, lipids, proteins, simple sugars, starches, water, hydrocarbons, ash, and other components with numerous functional groups are also comprised. 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 & Mazzoni, 2014).



Figure 7: Banana peel wastes (Deithorn & Mazzoni, 2014)

2.4 Activation of Carbon from Various Precursors

2.4.1 Activated Carbon from Sugar Cane to Eliminate Rhodamine-B

The primary objective of the study is to investigate the feasibility of developing an efficient adsorbent from agricultural waste through chemical activation using phosphoric acid (H₃PO₄) and potassium hydroxide (KOH) as activating agent. In addition, this study also comprises the adsorption capacity of activated carbon produced from bagasse pith by removal of dye from aqueous solutions, especially in wastewater treatment systems. The bagasse pith collected from sugar cane is used as precursor for carbon activation using one-step activation method which requires activation agent such as phosphoric acid (H₃PO₄) or potassium hydroxide (KOH). Once the bagasse pith is completely soaked, they are dried in oven at 80°C overnight and then carbonized in tubular electric furnace which is open at both ends. The carbonization was done at 500°C for 80 minutes in limited air condition. The pore characteristics and surface area of the activated carbon was studied using nitrogen adsorption-desorption in a gas sorption analyser at -196°C.

(RhB) as adsorbate and activated carbon derived from bagasse pith as adsorbent (Gad & El-Sayed, 2009).

The factors determining the adsorption capacity was identified which include:

- i. Physical nature of adsorbent (activated carbon) pore characteristics
- ii. Features of adsorbate (dye)
- iii. Solution circumstances

The pores of activated carbon derived from bagasse pith were characterized using N_2 adsorption with adsorption isotherms developed accordingly. The adsorption isotherms are illustrated below.



Figure 8: Nitrogen adsorption/desorption isotherm on bagasse pith, H₃PO₄ activated carbon (H.M.H.Gad & A.A.El-Sayed, March, 2009)

Activated carbon derived from bagasse pith shows a blend of Type I and II of IUPAC isotherm characteristics comprising micropores and mesopores. The summary of the pore characteristics of activated carbon is tabulated.

IUPAC Classification of Pores	Pore Diameter
Macropore	Larger than 50 nm
Mesopore	50 - 2 nm
Micropore	Smaller than 2 nm
Super-micropore	0.7 - 2 nm
Ultra-micropore	0.7 - 0.4 nm
Sub-micropore	Smaller than 0.4 nm

Table 1: IUPAC Classification of Pore Sizes (H.Gad & A.El-Sayed, 2009)

2.4.2 Activated Carbon from Coconut Shell for Carbon Dioxide Removal

According to a research done by N.A. Rashidi, S.Yusup and A.Borhan (2014) entitled, Novel Low-Cost Activated Carbon form Coconut Shell and Its Adsorptive Characteristics for Carbon Dioxide, activated carbon derived from carbonaceous material, mainly agricultural wastes such coconut shell can be possible excellent adsorbent in the effort for removing gaseous particles such as carbon dioxide. The objective of the study is to produce the microporous activated carbon resulting from coconut shell and study its applicability for carbon dioxide, CO₂ gas adsorption. There are commonly two types of activation methods that can be used which are chemical activation and physical activation. However, due to high energy consumption and long operating hours for double physical activation as well as waste disposal issues for chemical activation, a new activation method was utilized in order to prepare activated carbon, one-step CO₂ activation involves single activation technique under the carbon dioxide flow, without undergoing the carbonization process under an inert atmosphere. The carbon dioxide serves dual purposes, that is, as a purging gas to eliminate all volatile substances as well as an activation medium to react with carbon structures and develop porosity. The pore characterization was done using the nitrogen, N₂ adsorption isotherm of activated carbon, which was found out to follow the Type I isotherm that corresponds to microporous material. In addition, the sharp increase in N₂ adsorbed in the lower relative pressure region is due to the micropore filling. The micro-porosity in the activated carbon is very useful for gas adsorption process, provided that the gases molecular diameter is between the ranges of 0.4 mm - 0.9 mm.

At raised temperature, the adsorbed molecules on the carbons' surface achieve sufficient energy to overcome the van der Waals bonding and interchange to the gas phase over again. It was also observed that nitrogen adsorption capacity is lower compared to carbon dioxide regardless of the temperature. Carbon dioxide possesses smaller molecular diameter which is approximately 0.33 nm. This enables better diffusivity into the porous network in the samples compared to nitrogen which has a molecular dimension of 0.364 nm. Besides that, commercial activated carbon reports higher adsorption capacity compared to the synthesized activated carbon due to its bigger surface area which provides larger site for adsorption to take place (Rashidi, Yusup & Borhan, 2014).

2.4.3 Activated Carbon from Petroleum Residue to Eliminate CO₂

A sorbent is certified to be industrially keen when it is porous, comprises of high adsorptive capacity as well as selectivity towards carbon dioxide (CO_2). High pressure working unit, Pressure-Swing Adsorption (PSA) is used to give a better understanding on critical porous structure of activated carbon under extreme conditions. Activated carbon based on petroleum residue correlate a vastly developed micro- and meso-porous structure which offers great adsorption capacity, extraordinary selectivity towards CO_2 with an easy regeneration under trivial surroundings (Casco et.al, 2014).

Manometric equipment that is totally automated was used to study highpressure analysis. Type I isotherm was exhibited by low ratio samples, thus demonstrating the existence of a fine micropore size network. Increase in concentration of activating agent results in progress of novel micropores as well as broadening of existing pores. Sample activated with lowest ratio of potassium hydroxide (KOH) signifies highest amount of carbon dioxide adsorbed. Conversely, this inclination is totally inverted beyond 4.5 MPa (Casco et.al, 2014). Consequently, CO₂ adsorption at atmospheric and high-pressure restores tiny micropores to enhance the adsorption capacity especially at atmospheric pressure whereas larger micropores and also small mesopores manage the adsorption at higher pressures (Casco et.al, 2014).

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2.4.4 Comparison between Chemical Activation and Physical Activation

Chemical activation is preferred compared to physical activation with regard to low temperatures and short time for activation (Gad & El-Sayed, 2009). Physical activation takes place with hot gases as activating agents like carbon dioxide and steam. On the other hand, carbon yield is increased by using chemical agents which act as dehydrating agents contributing to pyrolytic disintegration and hinder tar production (Gad & El-Sayed, 2009). In addition, oxygenated surfaces of activated carbon are formed due to chemical activation method. Therefore, there are significant advantages of chemical activation compared to physical activation with respect to greater yield with enhanced surface area as well as improved porous formation (Gad & El-Sayed, 2009).

2.5 Factors Contributing to Development of Pores

In a study by Y. Örküna et.al entitled Influence of Temperature and Impregnation Ratio of H₃PO₄ on the Production of Activated Carbon from Hazelnut Shell, chemical activation of carbon is utilized with the usage of phosphoric acid (H₃PO₄). Operating parameters that were studied are concentration of activating agents used, activating temperature and activation time in interest to study the effects of operating circumstances on evolutions of pore volume and pore size of activated carbons derived from hazelnut shell (Örkün, Karatepe, & Yavuz, 2012). The particle sizes of hazelnut that were impregnated are between 0.85 mm and 1.0 mm. Three impregnation ratios that were prepared are 35wt%, 50wt% and 60wt%. The BET surface areas (S_{BET}), micropore volumes (V_{mi}) and mesopore volumes (V_{me}) of the activated carbon samples were calculated from the N₂ adsorption isotherms using the BET equation. The result shows Type I isotherm which suits the Brunauer, Deming, and Teller (BDT) classification, representing unimolecular adsorption (Örkün, Karatepe, & Yavuz, 2012). It was stated that this type of isotherm applies in adsorbent with small pore sizes of micropores range. The pore size and pore volume enhance until 450°C and shows reduction in pores due to an rise in temperature. Longer activation time up to 3 hours increases and widens the pores which prove that prolonged carbonization is desired to ensure complete development of porosity at given temperature (Örkün, Karatepe, & Yavuz, 2012).

2.6 Adsorption Capacity of Activated Carbon for Heavy Metals

According to *Biosorption of Heavy Metal Ions from Industrial Waste Water by Banana Peel Based Biosorbent* written by P.K. Hoong, it was proven that the optimum activation temperature would be at 400°C as any temperature higher than that yields poor results of activated carbon with low yield of S_{BET} . In addition, samples with larger particle size also demonstrate lower S_{BET} compared to smaller particles due to smaller surface area available for activation during impregnation. Furthermore, high impregnation ratio is deduced not to be desirable for zinc chloride unless high activation temperature used.

Sample A20 has the largest S_{BET} among all the other sample analysed. Its adsorption isotherm evidently follows Type III which is related to the physical adsorption of gases whereby the adsorbent-adsorbate interaction is weak compared to the adsorbate-adsorbate interaction.

In the adsorption capacity test, sample A20, which the banana peel is impregnated with phosphoric acid, ratio 1:1 and carbonized at 400° for 2 hours is selected to be used in the batch adsorption of crude oil and heavy metals, which are Zn, Pb and Cu. The result of the test shows that the samples are effective in removing oil & grease particles and the selected heavy metals from their respective solution, which could be attributed to the high value of S_{BET} .

CHAPTER 3

METHODOLOGY

3.1 Key Milestone



3.2 Experiment Methodology

3.2.1 Banana peel sample preparation

Banana peel wastes of weight approximately 6 kg were collected from fried banana stalls nearby in use of this project work. They are thoroughly washed in order to remove muck and cut into smaller pieces. The washed banana peels are kept in open atmosphere to evaporate dampness from its surface and dried in microwave oven at 80°C for two nights to remove excess moisture. Once the banana peels are completely dry, they are grinded into smaller pieces until become powder form. After grinding, the banana peels are sieved to separate particles of different size and kept in air tight container for activation (Rashidi, Yusup, & Borhan, 2014).



Figure 9: Powdered banana peel 0.25 mm Figure 10: Powdered banana peel 3.00mm

3.2.2 Activation of carbon in banana peel

The parameters which are manipulated in the activation phase of the activated carbon derived from banana peel are the impregnation ratio, carbonizing temperature as well as duration of carbon activation. The activation agent used in this study is potassium hydroxide (KOH). Potassium hydroxide is chosen in this study based on the previous researches conducted on similar lignocelluloic precursor and the results of using the above mentioned chemical shows better pore formation in the activated carbon compared to available chemicals (Ademiluyi & David-West, 2012), (Borhan & Kamil, 2012), (Diao, Walawender, & Fan, 2002), (Toles, Marshall, Johns, Wartelle, & McAloon, 2000). The impregnation ratio studied in this project is 1:1, 1:2, and 1:3. Impregnation ratio is the dry weight of powdered banana peel, W_{BP} divided by the dry weight of activation agent used, W_{KOH} .

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

About 10g of banana peel bio-sorbent is soaked in 100 mL of activating chemical agent overnight. This procedure is to ensure that the reagents are completely soaked and adsorbed into banana peel powder. The beaker containing the mixture of potassium hydroxide (KOH) and powdered banana peel is covered with aluminium foil to avoid contamination or evaporation of the chemical agent.



Figure 11: Banana peel powder soaked in KOH

The banana peel is then filtered from the chemical agents (Rashidi, Yusup, and Borhan, 2014). The residue, impregnated banana peel powder, is then carbonized in tubular furnace under steady flow of nitrogen gas (N₂). The ranges of temperature set for the carbonization process are 400°C, 500°C and 600°C for one hour and two hours durations. This is the point, where the banana peel powder turned into activated carbon (Rashidi, Yusup, and Borhan, 2014).



Figure 12: Filtration of soaked banana peel

Once the samples burned in furnace have cooled down to room temperature, they are washed repetitively to ensure the samples are free from chemical traces. This measure is to ensure there are no traces amounts of impurities which may trap in the pores of activated carbon and eventually, interrupt the adsorption activity of carbon dioxide later.



Figure 13: Banana peel before carbonization



Figure 14: Banana peel after carbonization

Lastly, the samples are placed in respective tubes with correct labelling and stored in an impenetrable container to prevent humidity contaminating the samples. Each set of parameters is used for two particle sizes of banana peel powder, which are 0.25mm and 3.00mm. In total, there are 36 samples to be prepared for all the combination of parameters under study.

The preparation condition and the status of each sample can be found in APPENDIX while the breakdown of sample preparation is illustrated in the figure below:



Figure 15: Steps in Preparing Activated Carbon

3.3 Characterization of Sample

In order to analyse and characterize the samples accordingly, several analysing equipment have been utilized. First and foremost, Field Emission Scanning Electron Microscope (FESEM) was used to give magnified images of the surface of activated carbon derived from banana peel. This is to ensure the formation of adsorption sites, also known as pores. FESEM with ultra-high-resolution imaging is designed to fulfil the requirements of analysing up to nano scale surface structure and morphology of solids. The characteristic X-rays generated are used for identification of different elements present in the specimen by energy dispersive spectrometer (EDS). This EDS technology is used to detect the element composition in the samples before and after activation in order to study whether the carbon content in activated carbon of banana peel is adequate for commercial production of activated carbon.



Figure 16: Field Emission Scanning Electron Microscope (FESEM)

In addition, the pore size distribution, specific surface area as well as the porosity of the samples is determined by the nitrogen adsorption-desorption isotherms characterized by the Micromeritics ASAP 2020. This equipment operates using nitrogen gas as adsorbate while being degassed at 350° C for 4 hours. The specific surface area of the bio-sorbent samples is estimated by the Brunauer-Emmett-Teller (BET) method using the N₂ 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 strongly desired in the sample to be qualified as activated carbon (Borhan & Kamil, 2012).



Figure 17: Micromeritics ASAP 2020

Last but not least, High Pressure Volumetric Analyser (HPVA-II) was used to study the carbon dioxide adsorption capacity on activated carbon derived from banana peel. 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 18: HPVA-II for Carbon Dioxide Adsorption Analysis



Figure 19: HPVA System Schematic Diagram

1. Tools and Equipment

Table 2: Lists of tools a	and equipment
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No	Equipment	Uses
1	Field Emission Scanning	To provide magnified view of the
	Electron Microscope	activated carbon structure derived from
	(FESEM)	banana peel
2	Surface Area Analyzer and	To determine the pore size distribution,
	Porosimetry Systems	specific surface area and porosity of the
	(MICROMERITICS ASAP	banana peel activated carbon through
	2020)	nitrogen adsorption-desorption isotherms
3	Drying Oven	To remove moisture from banana peel
4	Tubular Furnace	To carbonize banana peel into activated
		carbon
5	HPVA II Analyzer	To test the adsorption capacity of carbon
		dioxide on prepared activated carbon

2. Substance and Chemicals

Table 3: Lists of substances and chemicals used

No	Materials	Purpose
1	Banana Peel	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

No	Detail		Week												
110			2	3	4	5	6	7	8	9	10	11	12	13	14
1	Title Selection and Allocation														
2	Submission of Title Selection Form														
3	Title and Supervisor Allocation														
4	Preliminary Research Work														
5	Preparing Extended Proposal														
6	Submission of Extended Proposal														
7	Proposal Defence														
8	Project Work Continues														
9	Submission of Interim Draft Report														
10	Submission of Final Interim Report														

No	Detail		Week												
	Douin	15	16	17	18	19	20	21	22	23	24	25	26	27	28
	Project Work Continues														
11	• Samples sent for characterization														
11	• Test adsorption capacity with HPVA II analyser														
	• Collect results and discuss the result obtained														
12	Submission of Progress Report														
13	Pre-SEDEX Presentation														
14	Submission of Technical Paper														
15	Submission of Dissertation														
16	Oral Presentation														
17	Submission of Final Project Report														

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)

Element	Before Carbon	ization (RAW)	After Carbonization (A1			
	Weight %	Atomic %	Weight %	Atomic %		
Carbon, C	60.87	67.85	58.18	72.55		
Oxygen, O	23.74	25.35	25.19	23.57		
Silicon, Si	0.43	0.26	0.63	0.34		
Potassium, K	14.96	6.54	15.37	3.27		
Chlorine	-	-	0.63	0.27		
Total	100	100	100	100		

Table 4: Elemental Composition for RAW Sample and Sample A10

The average yield based on all the samples that have been converted into activated carbon is **40.78%**. The yield for each sample prepared is shown in APPENDIX. It is distinguished that although the weight of banana peel powder used for all samples is fixed at 10g, there are some loss in banana peel powder mass during the transfer from the filter paper to the crucible for carbonization after the banana peel powder is impregnated by the chemical agents. Therefore, the tabulated mass of banana peel powder in APPENDIX shows less than 10g.

For the analysis using the Energy Dispersive X-ray (EDX) Spectroscopy, it is determined that the carbon content (weight) of raw banana peel sample is **60.87%** which indicates that banana peel is appropriate to be transformed into activated carbon since its carbon content falls within the desirable range (50% - 80%) (Wu, Tseng, & Hu, 2005).

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 A10 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 oxygen, O content is also higher in sample A10. Both of these observations can be attributed 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.





Based on the FESEM imaging, Figure 20 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 21 is the 3000 times magnification on sample A10. The image clearly shows that pores have already been formed on the sample at 400°C and two hours of carbonization. More well-structured pores are observed on the surface morphology of sample A6 as shown in Figure 22, but at 500°C of carbonization temperature for one hour, the porous wall are broken slightly. However, Figure 23 shows the effect of implying excessive heat during carbonization, which is 600°C for two hours. 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.

4.2 Surface Area and Porosity Analysis

Sample	ID	A.Temperature	A.Period	S _{BET}	V _T	D(nm)
Label	IK	(°C)	(hours)	(m^2/g)	(cm^3/g)	D (IIII)
A1	1:1	400	1	31.4678	0.01593	4.5137
A10	1:1	400	2	260.3841	0.01436	0.2508
A19	1:1	300	2	258.2218	0.01297	0.2078
A6	1:3	500	1	3.7594	0.00472	1.9763
A8	1:2	600	1	2.5643	0.00294	0.7908
B11	1:2	400	2	40.0171	0.00705	2.8672
B15	1:3	500	2	1.6729	0.00397	1.0927
B18	1:3	600	2	10.5239	0.00206	0.2746
B2	1:2	400	1	7.8026	0.01273	6.0024

Table 5: Surface area and porosity results for selected samples

The table above shows the resulting surface area of the pore formed on activated carbon derived from banana peel at different preparation conditions. Based on the results, it shows that 400°C is the maximum carbonization temperature for banana peel based activated carbon and any higher activation temperature than 400°C produces deprived results.

Samples B2, B11, B15, and B18 are all carbonized at 400°C, 500°C and 600°C respectively, yield low S_{BET} with the exception of sample A10, which is carbonized at 400°C for 2 hours, but yields comparatively higher S_{BET} . In addition, group B samples which are of bigger particle size (3.00mm) during grinding, yields smaller surface area, S_{BET} compared to that group A samples with smaller particle size (0.25mm). This phenomenon occurs because larger particle size exposes less surface area for activation during impregnation phase, thus resulting in smaller S_{BET} .

Commonly, pore broadening will occur due to the rise in activation temperature and the impregnation ratio of KOH. Due to carbon gasification enhancement with elevated KOH ratio, 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). Samples containing large compositions of potassium element explain that high impregnation ratio yields potassium carbonate, K₂CO₃ and potassium oxide, K₂O during pyrolysis. Therefore, we can conclude that higher impregnation ratio forms insulating layer which covers the particles and reduces the interaction of pores and surrounding environment, thus resulting in lower activation rate (Mopoung, 2008).

Besides, by comparing between samples A6 and A8, results show that high impregnation ratio is not desired for KOH activation as it reduces the efficiency of pore formation with smaller surface area compared to those which are impregnated at smaller ratio. The highly microporous activated carbon shifts to a different form, where the mesopores become governing in whole pore size distribution, especially exceeding the certain limit of activating agent (Örkün, Karatepe, and Yavuz, 2011). It is also noticeable that as the concentration of activating agent crosses its limit, there will be a significant change in pore size developments due to high composition of phosphorous which will react with lignocellulosic contents during activation as well as impregnation phases (Örkün, Karatepe, and Yavuz, 2011). In addition, the higher the impregnation ratio, the higher the amount of dehydrating agent which further promotes pore formation. Unfortunately, when the limit is exceeded, again it reduces the pore formation (Cao, .Xie, Liv, & Bao, 2006). Hence, the excessive activating chemical decomposes into water resulting in gasification under high temperature (Hoong & Borhan, 2013).

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 A10 yields the biggest surface area, S_{BET} with area 260.3841 m²/g with pore diameter 0.2508 nm of sub-microporous group. Since sample A10 exhibits the best result among others, thus, this sample was utilized to study the adsorption capacity of carbon dioxide.

4.3 Nitrogen Adsorption-Desorption Isotherm



Figure 24: Nitrogen Adsorption-Desorption Isotherm for Selected Samples

Figure 24 above 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 24, samples A6, A8, A19 and B11 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 meso- to macro- porosity. They also have a mixture of strong and weak adsorbate-adsorbent interaction. Since these samples reveal smaller surface area with microporosity range of pore diameter, hence their isotherms tend to follow TYPE II and TYPE III isotherms which supposing describes the isotherm of larger porosity. Figure 24 also illustrates the adsorption isotherm for the largest surface area yielding sample, that is, sample A10. Adsorption isotherm of A10 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 sub-microporosity group with pore diameter, 0.2508 nm.

4.4 Carbon Dioxide Adsorption Analysis



Figure 25: Weight percentage of carbon dioxide adsorbed

Figure 25 shows the amount of carbon dioxide adsorbed in weight percentage with increasing pressure. The adsorption test is carried out using HPVA II analyser at optimum temperature of 25°C. The amount of carbon dioxide adsorbed increases with increasing pressure, proving that activated carbon derived from banana peel is proficient in removing carbon dioxide gas with microporous pore formation.



Figure 26: Adsorption capacity of carbon dioxide on activated carbon derived from banana peel

The adsorption isotherm in Figure 26 evidently have the similar shape to Type I isotherm, that is, adsorbent pores with microporous structure. The adsorption isotherm shows decreasing trend with inclined temperature. Thus, carbon dioxide adsorption is clearly ideal at lower temperature because it is an exothermic process, whereby heat is released (I.A.A.C.Esteves, et.al, 2008).

Type I isotherm implies the features of microporous solid matters with moderately lesser fraction on the external surface. Moreover, typical microporous pores are exposed to surface with mostly being micropores in inner side. They do not leave empty spots for further adsorption once the adsorbates have occupied the pores. Besides that, Type I isotherm is also known as pseudo-Langmuir isotherm as it portrays single layer adsorption well.

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 26. 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%.



Figure 27: Adsorption isotherm of activated carbon derived from petroleum pitch (M.E.Casco, et al., 2013)



Figure 28: Adsorption isotherm of activated carbon derived from carbon

(B.Guo, et.al., 2006)



Figure 29: Adsorption isotherm of activated carbon derived from coconut (L.I.Fenrong, et.al., 2010)



Figure 30: Adsorption isotherm of activated carbon derived from coal, extruded carbon (I.A.A.C.Esteves, et.al., 2008)



Figure 31: Adsorption capacity of carbon dioxide on activated carbon derived from *Eucalyptus camaldulensis* wood at different temperatures (A.Heidari, et.al,

2013)

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 31. 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 (A.Heidari, et.al, 2013). Therefore, it is reasonable that adsorption test for carbon dioxide on banana peel based activated carbon is studied at low temperature of 25°C.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 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 is established to 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 400°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 A10 proved to be the best sample among others with its outstanding BET surface area as well as nitrogen adsorption-desorption isotherm indicating meso- and micro-porosity. 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 and contact period of the adsorption process. The sample with the highest S_{BET} from this project (sample A10) 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 32: BET Adsorption Isotherms (M.J.Kim & G.H.Chea, 2012)

No	Activating Agent	Impregnation Ratio	Activating Temperature (°C)	Activation Time (hour)	Impregnation	Carbonization	Weight before carbonization (g)	Weight after carbonization (g)	Yield (%)
1	КОН	1:1	400	1	Yes	Yes	8.72	3.81	43.7
2	КОН	1:2	400	1	Yes	Yes	9.46	2.98	31.5
3	КОН	1:3	400	1	Yes	No	7.74	-	-
4	КОН	1:1	500	1	Yes	No	9.05	-	-
5	КОН	1:2	500	1	Yes	No	7.34	-	-
6	КОН	1:3	500	1	Yes	Yes	7.80	2.25	28.8
7	КОН	1:1	600	1	Yes	No	7.45	-	-
8	КОН	1:2	600	1	Yes	Yes	6.32	4.87	77.1
9	КОН	1:3	600	1	Yes	Yes	9.50	2.73	28.7
10	КОН	1:1	400	2	Yes	Yes	9.00	4.65	51.7
11	КОН	1:2	400	2	Yes	No	7.58	-	-
12	КОН	1:3	400	2	Yes	Yes	9.70	5.90	60.8
13	КОН	1:1	500	2	Yes	Yes	7.56	2.25	29.8
14	КОН	1:2	500	2	Yes	Yes	6.92	1.14	16.5
15	КОН	1:3	500	2	Yes	No	8.76	-	-
16	КОН	1:1	600	2	Yes	No	8.90	-	-
17	КОН	1:2	600	2	Yes	Yes	8.50	2.95	34.7
18	КОН	1:3	600	2	Yes	Yes	7.21	2.25	31.2

Table 6: List of samples Group A (particle size = 0.25mm) with respective preparation conditions

No	Activating	Impregnation	Activating	Activation	Impregnation	Carbonization	Weight before	Weight after	Yield
110	Agent	Ratio	Temperature (°C)	Time (hour)	mpregnation	Carbonization	carbonization (g)	carbonization (g)	(%)
1	КОН	1:1	400	1	Yes	No	8.52	-	-
2	КОН	1:2	400	1	Yes	Yes	9.82	3.95	40.2
3	КОН	1:3	400	1	Yes	No	7.54	-	-
4	КОН	1:1	500	1	Yes	No	7.98	-	-
5	КОН	1:2	500	1	Yes	No	9.46	-	-
6	КОН	1:3	500	1	Yes	No	8.78	-	-
7	КОН	1:1	600	1	Yes	No	6.78	-	-
8	КОН	1:2	600	1	Yes	No	9.45	-	-
9	КОН	1:3	600	1	Yes	Yes	7.42	1.56	21.0
10	КОН	1:1	400	2	Yes	No	7.89	-	-
11	КОН	1:2	400	2	Yes	Yes	9.90	7.89	79.7
12	КОН	1:3	400	2	Yes	No	9.89	-	-
13	КОН	1:1	500	2	Yes	No	6.89	-	-
14	КОН	1:2	500	2	Yes	No	8.75	-	-
15	КОН	1:3	500	2	Yes	Yes	6.55	2.46	37.6
16	КОН	1:1	600	2	Yes	Yes	9.67	4.23	43.7
17	КОН	1:2	600	2	Yes	No	7.56	-	-
18	КОН	1:3	600	2	Yes	Yes	7.89	2.88	36.5

Table 7: List of samples Group B (particle size = 3.00mm) with respective preparation conditions

RAW

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

- C CaCO3 1-Jun-1999 12:00 AM
- O SiO2 1-Jun-1999 12:00 AM
- Si SiO2 1-Jun-1999 12:00 AM
- K MAD-10 Feldspar 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
СК	60.87	67.85
OK	22.74	25.25
UK	23.74	25.35
Si K	0.43	0.26
K K	14.96	6 54
КК	14.90	0.54
Totals	100.00	





Electron Image 1





A10

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 4

Standard :

- C CaCO3 1-Jun-1999 12:00 AM
- O SiO2 1-Jun-1999 12:00 AM
- Si SiO2 1-Jun-1999 12:00 AM
- CI KCI 1-Jun-1999 12:00 AM
- K MAD-10 Feldspar 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
СК	58.18	72.55
ОК	25.19	23.57
Si K	0.63	0.34
CI K	0.63	0.27
КК	15.37	3.27
Totals	100.00	
	1	



200µm Electron Image 1





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