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ACTIVATION OF CHARCOAL BY PHOSPHORIC ACID IMPREGNATION FOR HIGH YIELDING ACTIVATED CARBON

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Dissertation submitted in partial fulfillment of the requirements for the

Bachelor of Chemical Engineering (Hons)

Sept 2014

Universiti Teknologi PETRONAS,

Bandar Seri Iskandar,

31750 Tronoh,

Perak.

CERTIFICATION OF APPROVAL

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Approved by,

____

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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.

(Kanaheswaree Rajendran)

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Abstract

The main aim of this research is to study the prospective of using charcoal as a potential adsorbent for eliminating carbon dioxide from polluted air in the atmosphere. This project works highlights on the constraints involved in the preparation of activated carbon from charcoal such as activating agent, activating temperature, ratio of impregnation. The carbonization process allows forming an active site for adsorption. Micromeritics ASAP 2020 and Field Emission Scanning Electron Microscope (FESEM) 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 (CO_2). The samples were also analyzed for its elemental composition before and after carbonization using FESEM which results in 29.6 wt% and 49.2 wt% of carbon content respectively. Sample A2 was identified to yield activated carbon with largest surface area, S_{BET} of 415.6512 m^2/g and pore diameter, 17.7 nm which falls in mesopore category. In addition, the activated carbons developed from charcoal are also verified for their adsorption capacity with harmful greenhouse gas such as carbon dioxide using HPVA II analyzer.

1.0 Introduction

1.1 Background

This project is basically about removing Carbon Dioxide (CO_2) to reduce the level of CO_2 level in the atmosphere by activating carbon using charcoals. There are few technologies were developed to reduce CO_2 from atmosphere such as bio-energy with carbon capture and storage, direct air capture, ocean fertilization and enhanced weathering as well as adsorption[1]. Any materials (animal, plant, or mineral origin) with high concentration of carbon can be simply changed into activated carbon using chemical or physical activation methods. The most common raw materials are wood, charcoal, nut shells, fruit pits, brown and bituminous coals, lignite, peat, bone and paper mill waste (lignin), synthetic polymers like PVC, are used for manufacturing of activated carbon [2]. Since earliest times, charcoal has been used for a large range of purposes especially as a purifier. A wide range of organic compounds dissolved or suspended in gases and liquids is been adsorbed easily by charcoal. Adsorbents are usually porous solids, and adsorption occurs mainly on the pore walls inside particles which allow for remarkable adsorption capacities [2]. Activating charcoal also leads to activation of Carbon atom in charcoal which eventually increases its effectiveness as a filter. Thus, charcoal was chosen as an adsorbent for this project as it is easy to obtain from market, through chemical activation using phosphoric acid. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material [3]. A few parameters are taken into account to obtain a better result. The parameters that will be studied in this experiment are the size of the particles used, impregnation time, temperature, specific surface area, total pore volume and average pore diameter.

Figure 1.1.1: The Figure shows a carbon dioxide molecule

1.2 Problem statement

Carbon dioxide (CO_2) is a naturally occurring chemical compound composed of 2 oxygen atoms each covalently double bonded to a single carbon atom. It is a gas at standard temperature and pressure and exists in Earth's atmosphere in this state, as a trace gas at a concentration of 0.04 per cent (400 parts per million, ppm) by volume, as of 2014 [[4]. Burning of carbon-based fuels since the industrial revolution has rapidly increased the concentration of carbon dioxide, leading to global warming. Carbon dioxide now an important greenhouse gas and it is also a major source of ocean acidification since it dissolves in water to form carbonic acid which is a weak acid as its ionization in water is incomplete [4]. Scientist at National Oceanic & Atmospheric Administration's (NOAA) Earth Research Laboratory calculates the Annual Greenhouse Gas Index (AGGI) each year about 80 sites around the world [4].

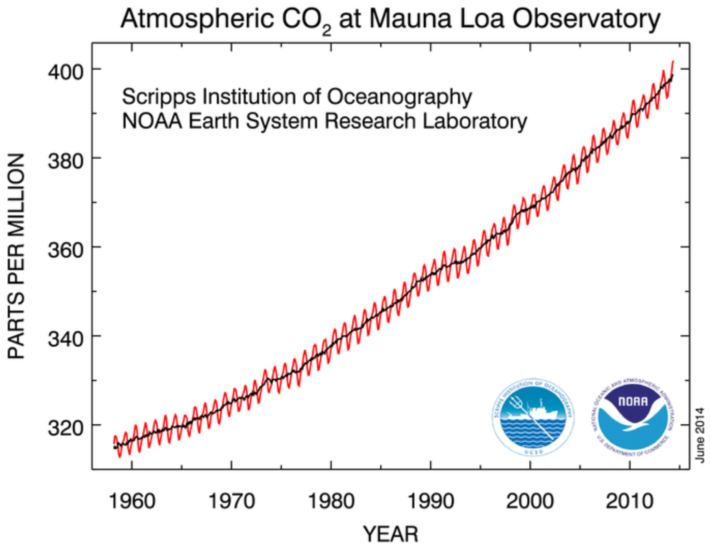


Figure 1.2.1: The graph shows recent annual mean growth gate measured at Mauna Loa, Hawaii [4]

Technologies were developed in order to reduce carbon dioxide concentration from Earth's atmosphere [1] and to produce an eco-friendly environment by the scientist. Technologies are such as bio-energy with carbon capture and storage, biochar (another name for charcoal), direct air capture, ocean fertilization and enhanced weathering. One of the most economically and environmentally preferred technology is adsorption because the other technologies tends to result in negative emission where it removes the greenhouse gas, carbon dioxide, permanently from Earth's atmosphere [5]. Charcoal is a form of carbon processed to have small, low-volume pores that increase the surface area available for adsorption. Due to its high degree of micro porosity, just one gram of activated carbon has a surface area in excess of 500 m^2, as determined by gas adsorption. An activation of charcoal increases the porosity [6] and results in an efficient adsorption process.

1.3 Objectives

The objectives of this project are:-

- **To prepare activated carbon from charcoal by chemical activation method.**
  Activation of carbon atoms in the charcoal can be done by impregnating it in phosphoric acid and then heated in furnace at 200-500˚C for about 12 hours.

- **To investigate the effect of the size of the charcoal, impregnation ratio of phosphoric acid, time and temperature effects on the porosity size of activated carbon.**
  Crushed charcoal sample impregnated at different ratio of phosphoric acid and time to find a better result in adsorption of carbon dioxide

- **To analyze the characterization of the pores of activated carbon by studying their pore size and adsorption isotherms respectively.**
  Freundlich and Langmuir equation are the most popular empirical equations which are used to express the mathematic relationship between the quantities of impurity remaining in solution versus the quantity adsorbed. The nitrogen adsorption-desorption isotherms were used to determine specific surface area and total pore volume.

- **To test the prepared activated carbon**

  Prepared activated carbon was analyzed for its characteristics and abilities.

1.4 Scope of study

This project uses phosphoric acid as the activating medium for charcoal. The pores of activated carbon are then tested for their characteristics with nitrogen (N2) and carbon dioxide (CO2) gases. The experiment is repeated for different operating parameters:

- Ratio of charcoal and phosphoric acid
- Pore size of activated carbon
- Carbon dioxide adsorption capacity

2.0 Literature review

2.1 Charcoal

When wood is "carbonized" or "pyrolysed" under controlled conditions in a closed space such as a charcoal kiln remaining solid residue is charcoal. During carbonization or pyrolysis, the process is carried out, away from the oxygen or air to prevent the wood to ignite and burn down to ashes. The wood substance is forced to decompose without oxygen and produces a few products especially charcoal [7].

The pyrolysis process might continue up to seven days. The size of the kiln and the density and freshness of the wood determines the duration of the combustion. The stage of combustion is detectable by checking the color and amount of the smoke. During the pyrolysis process, the mud-worker is required to fill up the openings with clay in order to guarantee that the trapped heat inside the kiln is suitable for manufacturing charcoal [8].

Figure 2.1.1: The Figure shows a kiln during combustion process [8].

2.2 Activated Carbon

Activated carbon is a form of carbon treated to have small, low-volume pores that increases the surface area available for adsorption or chemical reactions. Activated carbon has a surface area in excess of 500 m^2 per gram due to its high degree of micro porosity, as analyzed by gas adsorption [6].

An activation level adequate for convenient application may be attained solely from high surface area and chemical treatment often enhances adsorption properties [6]. The surface area of the pore surface developed within the matrix of the activated carbon can be measured using nitrogen gas using Brunauer-Emmet-Teller (BET) method. Based on the principle, greater surface area tends to have greater number of adsorptive sites [6].

The pore size distribution of an activated carbon can be determined and it is an extremely useful way of understanding the performance characteristics of the material. The International Union of Pure and Applied Chemistry (IUPAC) define the pore size distribution as follows:

- Micropores: $r < 1$nm
- Mesopores: $1 < r < 25$nm
- Macropores: $r > 25$nm

- The macropores are used as the entrance to the activated carbon, the mesopores for transportation, and the micropores for adsorption [6].

2.3 Activating Methods

Activated carbon is broadly used in various practices as a cost-effective and effective adsorbent. The feed materials used and the method of activation differs the adsorption property of an activated carbon. The use of cost-effective material in producing activated carbon might generate a low cost product [9].

Activated carbon can be produced by chemical activation and physical activation. Density and the size of the material is taken into consideration before choosing the activation method. During physical activation method, the bulk of volatile matter is eliminated from the raw material by carbonizing it at extremely high temperatures (400-500˚C). Carbon is then subjected to oxidizing gases such as carbon dioxide or steam at 800-1000˚C for selective oxidation. There are high chances for carbon atom to be oxidized by atmospheric oxygen and produces carbon dioxide, so the product should be precisely handled during carbonizing and activating period. Steam and carbon dioxide act as mild oxidizing agents at 800-1000˚C [2]. The reaction of carbonizing and steam activation method as follows:

$$C_x(H_2O)_y \longrightarrow xC(s) + yH_2O \qquad \text{Carbonizing}$$

$$C(s) + 2H_2O \longrightarrow CO_2 + 2H_2 \qquad \text{Steam activation}$$

$$C(s) + CO_2 \longrightarrow 2CO \qquad \text{Activation by } CO_2$$

While in chemical method, the carbonization process and activation processes are carried out at the same time. Raw material is crushed and impregnated with chemical agent in a proper ratio (ratio can be manipulated), dried and carbonized up to 800˚C in vacuum or in the presence of inert gases to make sure that there is no air and oxygen during the carbonization process. The residual impregnating agent is removed by leaching with water once the carbonization has been completed.

An extremely active decolorizing carbon is obtained by a rapid process in high yield at a relatively low reaction temperature around 350-500˚C with chemical agent activation. This is to degrade the organic molecules during carbonization that avoid deposition of hydrocarbon on the carbon surface [2]. Powdered activated carbon has an extremely high ratio of area to volume, and since adsorption is a surface related process, so this rises its effectiveness, but also makes it slow to settle down and difficult to remove once it is added. Most decolorizing carbons are prepared by chemical activation.

The yield and quality of the prepared carbon depends on the preparation variables on were studied. The activation temperature, heating rate, activation time and impregnation rate of the chemical agent was important in determining the quality of activated carbon obtained [9].

2.4 Adsorption

Adsorption is the adhesion of atoms, ions, or molecules from a gas, liquid, or dissolved solid to a surface [11]. This process generates a film of the adsorbate on the surface of the adsorbent while absorption is a different process where the fluid (the absorbate) infuses or is dissolved by a liquid or solid (the absorbent) [11]. The term sorption encompasses both processes, while desorption is the reverse of it. Activated charcoal is good at trapping other carbon-based impurities ("organic" chemicals and toxins), as well as things like chlorine. There are also some other chemicals are not attracted to carbon at all such as sodium and nitrates, they just pass right through. This means that an activated charcoal filter is only capable of removing certain impurities while ignoring other impurities [10].

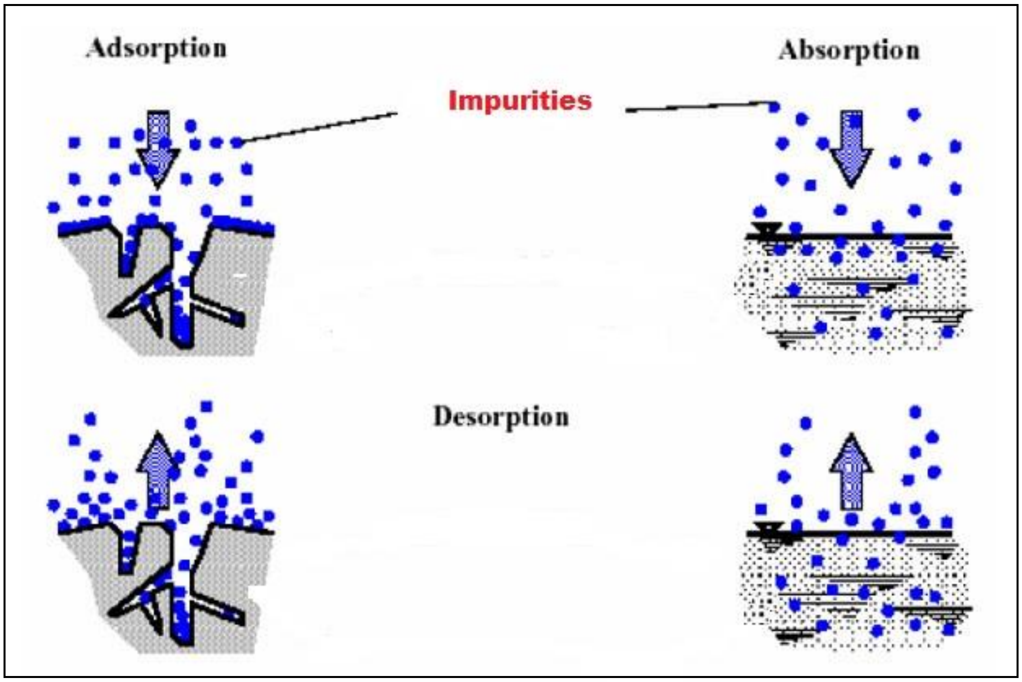


Figure 2.4.1: The Figure shows the difference between adsorption and absorption.

2.5 Materials & Methods

The materials that have been used in this project has been chose according the following considerations:

Item	Compositions
1. Banana peel	Lignin 7 - 15%, 6-9% protein and 20-30% fibre [17].
2. Charcoal	C > 96% -98%, O 1-2%, Mg, P, K, Na, maybe some Cl [16].
3. Sawdust	Moisture 4.8%, ash 0.21, CH_2OH 5.1%, Cellulose 58.2% and lignin 28.4% [18].
4. Apple pulp	Moisture 10-12%, ashes 3-5%, proteins 6-6.2% and pectin 9-10% [19].

Table 2.5.1 : Chemical composition of the selected items

From the analysis above, banana peel has less than 50% of carbon. Sawdust has carbon around 70% and apple pulp has 16% of carbon. While, charcoal has the highest percentage of carbon and carbon is the adsorbing agent according to the previous researches.

Physical activation method	Material was carbonized in the range of 600-900˚C in absence of oxygen by using inert gas such as Nitrogen. The raw material is then oxidized by exposing to steam or oxygen in the temperature range of 600-1200˚C [3].
Chemical activation method	The raw material is impregnated in strong

	base, acid or salt and then carbonized at lower temperature ranging 450-900˚C. It is convicted that carbonizing process and activating process occurs simultaneously [3].

Table 2.5.2 : Activation Methods

From the table above, chemical activation method is preferred because it can be achieved by using lower temperatures and in short period of time compared to physical activation method [3].

No	Author	Raw material	Experiment conditions	BET surface area (m^2/g)
1.	Krishnan, 2008	Sawdust	20% of K_2CO_3 solution was mixed with grinded sawdust. Carbonization was performed in the presence of steam in furnace at 600˚C [20].	683.3
2.	Wu et al, 2004	Wood	Wood was sealed in a ceramic oven and heated up to 550˚C. Steam generated from deionized water was drizzled into the oven for 3 hours at a rate of $3cm^3$/min. The product was placed in oven and heated for an hour at 780˚C [21].	1064
3.	Azry Borhan	Rubber seed	10 g or rubber seed shell was	1001.66

		shell	impregnated in 100 mL of KOH solution. It is then carbonized in fixed bed activation to produce activated carbon. Nitrogen gas was flowed for 30 minutes in the furnace with a temperature of 500˚C [22].	
4	Lua AC and T.Yang	Pistachio nut shell	With an impregnation ratio of 0.75 in $ZnCl_2$, an activation temperature of 400˚C, and a hold time of 1 h for pistachio nut shell [23].	1847.5

*Table 2.5.*3 *: Earlier Researches and The Descriptions*

From the earlier researches, it can be concluded that a few types of chemicals such as KOH, NaOH, $ZnCl_2$, K_2CO_3 and H_2PO_3 has been used to produce activated carbon through chemical activation method. Inorganic chemical such as zinc chloride and phosphoric acid and alkali metal hydroxide is used to degrade the organic molecules during carbonization that prevents the deposition of hydrocarbon on the carbon surface. Phosphoric acid is chosen to proceed this project since $ZnCl_2$ releases chlorine gas and into atmosphere and highly yieldable at a relatively low reaction temperature around 350-500˚C [2].

3.0 Methodology

3.1 Project Flow

Literature Review

- Preliminary research on the related topic regarding recent studies.
- Understanding the concept of adsorption, activated carbon, charcoal and carbon activation methods.
- Identified the variables of the project.
- Study on the procedure to use BET and FESEM.

Experiment

- Design experiment to develop activated carbon by impregnating charcoal for removal of carbon dioxide.
- Prepare chemicals and equipments required for the experimental work.
- One step activation process to be performed to activate carbon using vertical tubular furnace
- Thermal gravimetric analyzer will be used to study weight change of charcoal with respect to temperature.
- Micromeritics ASAP 2020 to be used for pore characterization.
- Volumetric adsorption studies will be carried out for carbon dioxide and nitrogen using HPVA II analyzer

Data Extraction

- Conduct experiment, collect data and analyze the data.
- Weight change of charcoal with respect to temperature.
- Pore characterization using IUPAC classification and surface area calculation using Brunauer-Emmett-Teller (BET) equation
- Adsoprtion capacity of carbon dioxide and nitrogen
- Results and discussions.

Conclusion

- Conclude the findings.
- Prepare interim report.

3.2 Experiment Methodology

1. Materials

Commercial charcoal will be used as a raw material in this project. Phosphoric acid will also be used to activate the carbon in the charcoal. Nitrogen gas and carbon dioxide gas will be used to determine the adsorption value of the activated carbon [14].

2. Preparation of sample

Charcoal was crushed finely and sieved to obtain particles around 0.25 mm. The sample was used as raw precursor [13]. Sample was rinsed and dried in a furnace for more than 24 hours to make sure there is no moisture in it. Dried sample was kept in a air tight container.

3. Activation of carbon

The charcoal was first soaked in different amounts of phosphoric acid for different duration of time at room temperature. The impregnation ratio defined as:

$$\text{Impregnation ratio} = \frac{\text{Weight of } H_3PO_4 \text{ in solution}}{\text{Weight of carbon}}$$

The chemical activation methods were conducted in a modified furnace with continuous output power. After impregnation, charcoal was filtered from phosphoric acid. There are three part in the apparatus used for the activation process which are the gas inlet, tubular reactor, and condenser. The furnace was flushed with highly purified nitrogen to maintain an inert condition in the furnace [13]. The activation process is estimated to accomplish at the 500˚C-600˚C for 4-5 hours.

4. Tools and equipments

No.	Equipments	Description
1.	Beaker	Impregnation of charcoal
2.	Conical flask	Mixing of phosphoric acid and charcoal.
3.	Weighing balance	Weigh the weight of charcoal before impregnating, after impregnating and after drying.
4.	Measuring cylinder	Measure volume of phosphoric acid.
5.	Filter funnel & filter paper	Filter impregnated charcoal.
6.	Furnace	Carbonization of charcoal.
7.	Grinder	Grind charcoal to different sizes.
8.	Oven	Dry impregnated charcoal.
9.	Thermal gravimetric analyzer	Study of weight change of charcoal with respect to temperature.
10.	Micromeritics ASAP 2020	Pore characterization.
11.	HPVA II analyzer	Volumetric adsorption was studied.

5. Range of variables

a) Study of weight change of charcoal versus temperature.

Temperature (˚C)	Weight of charcoal sample (g)
300	
400	
500	
600	

b) Study of surface area of pores formed with respect to impregnation ratio

Impregnation ratio	Surface area of pores formed (m^2)
1:1	
1:2	
1:3	

c) Study of carbon dioxide adsorption capacity with respect to temperature

Temperature (°C)	Carbon dioxide adsorption capacity (% weight)
50	
75	
100	

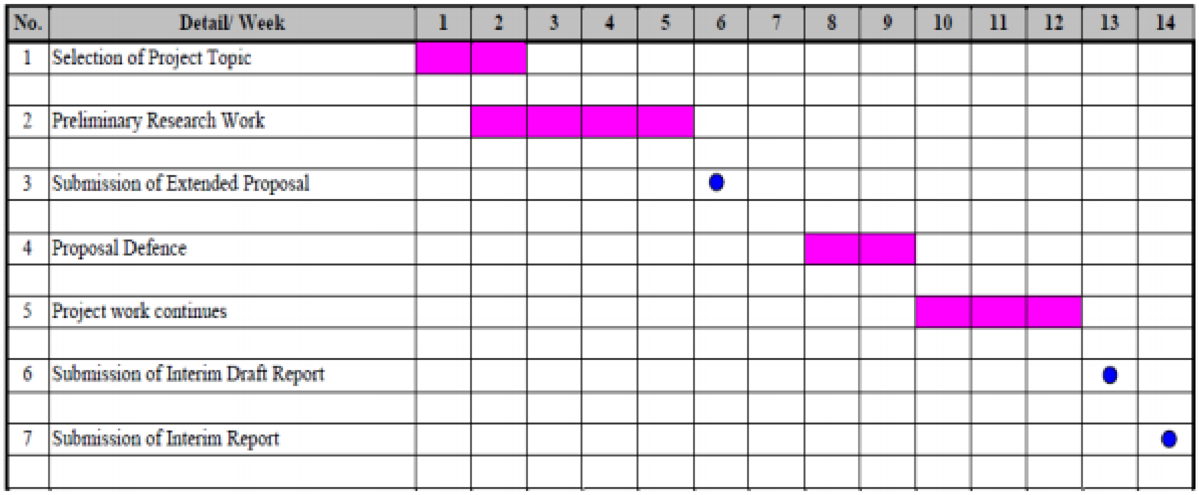
3.3 Proposed Experiment Procedure

Referring to the previous research work done by S.M. Yakout, and G. Sharaf El-Deen in their project, Characterization of activated carbon prepared by phosphoric acid activation of olive stones, the procedures for this experiment are as follows [15]:

1. Charcoal will be used as a precursor.
2. Grind, dry and sieve charcoal.
3. Obtain particle sizes comprised between1-2mm for the preparation of activated charcoal.
4. Measure 50g of charcoal and impregnate with phosphoric acid with ratio of 1:1, 1:2, and 1:3 for 1,2,3 and 4 hours each.
5. Carry out carbonization process in the furnace and calibrate the temperature of the furnace and nitrogen flow rate accordingly.
6. Cool down the system under nitrogen flow and wash the product obtained with distilled water until normal pH is achieved.
7. Dry the product in the oven at 120˚C.
8. Measure active carbon characterization by using nitrogen adsorption/desorption isotherms at an automatic adsorption instrument in relative pressure ranging from 0.1-0.5.
9. The mass change is charcoal is studied using AXSTAR TG/DTA 6300 thermal gravimetric with respect to temperature. The samples were heated from 25°C to 900° at heating rate of 20°C per minute under the presence of Nitrogen at a flow rate of 20mL/minute.
10. The pore structures of activated carbon are determined using Micromeritics ASAP 2020 with N2 adsorption-desorption.
11. Volumetric adsorption of carbon dioxide and nitrogen on the activated carbon derived from charcoal is analyzed using HPVA II analyzer.
12. The adsorption took place at three distinct temperatures, 50°, 75°C and 100°C with pressure interval of 0.1 bar.

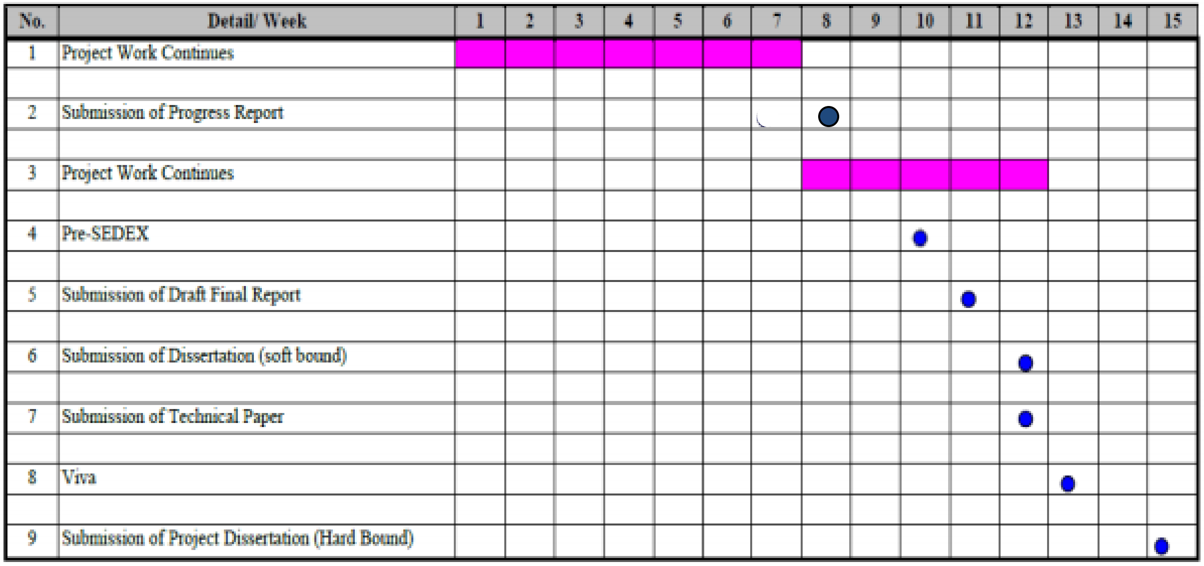
3.4 Gantt Chart & Key Milestone

Table 3.2.1: Gantt chart for FYP I



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 Defence								■	■					
5	Project work continues										■	■	■		
6	Submission of Interim Draft Report													●	
7	Submission of Interim Report														●

Table 3.2.2: Gantt chart for FYP II



No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues	■	■	■	■	■	■	■								
2	Submission of Progress Report								●							
3	Project Work Continues								■	■	■	■	■			
4	Pre-SEDEX										●					
5	Submission of Draft Final Report											●				
6	Submission of Dissertation (soft bound)												●			
7	Submission of Technical Paper												●			
8	Viva													●		
9	Submission of Project Dissertation (Hard Bound)															●

4.0 Results and Discussions

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 charcoal. The formula for the calculation of yield (Diao, Walawender, & Fan, 2002) is as follows:

$$Yield = \frac{M_{Ac}}{M_C} X\ 100\%$$

Whereby:

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

M_C = original mass of the carbon powder before carbonization (g)

Elemental Composition for the entire carbon sample is shown in the table below:

Element	Before the experiments		After the experiments			Yield (%)
	C (%)	O (%)	C (%)	O (%)	P (%)	
A1	29.6	70.4	29.9	72.1	-	1.2
A2	**29.6**	**70.4**	**51.0**	**49.0**	**-**	**72.30**
B1	29.6	70.4	29.8	70.2	-	0.67
E1	29.6	70.4	39.5	60.5	-	33.45
E2	29.6	70.4	48.4	48.5	3.1	63.54
F1	29.6	70.4	33.2	66.8	-	12.16
F2	29.6	70.4	35.4	64.5	-	19.60
J2	68.7	31.3	51.0	47.0	3.0	68.41

Table 4.1.1: Elemental Composition of Carbon samples before and after the carbonization process

Based on the elemental composition of carbon before and after, A2 yields 51.0% of carbon in the sample which results in a yield of 72.3% of carbon comparing with the initial sample. The sample A2 was 0.25mm sample which was carbonized at 500 ˚C. The ratio of sample: phosphoric acid for A2 is 1:10 and it yields a pore size of 17.7nm. The amount of carbon element in the elemental composition of FESEM shows that the pore size of the sample has become bigger which enables a bigger surface area for the sample to increase the adsorption activity.

In sample E2 and J2, there are some traces of Phosphorus which proves that the substance has been impregnated in a solution or material that contains Phosphorus, which is Phosphoric acid.

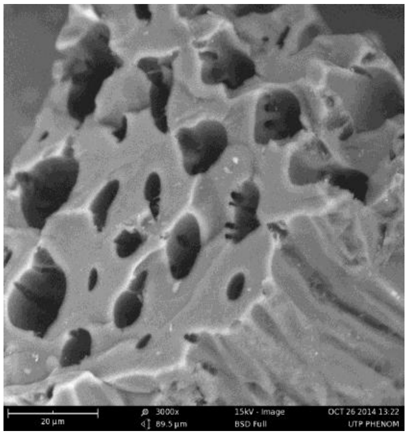


Figure 4.1.1: 0.25mm Pure Sample FESEM image

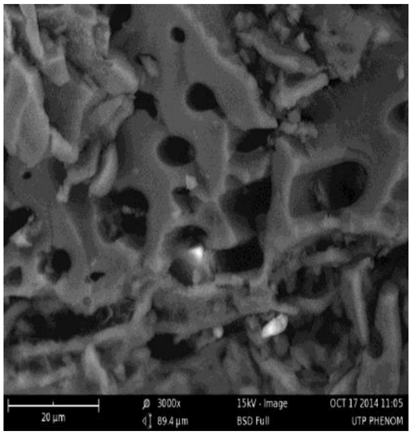


Figure 4.1.2: Sample A2 FESEM image

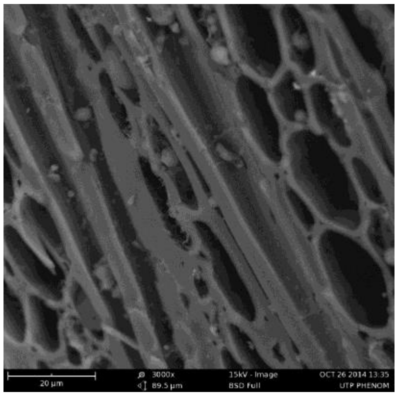


Figure 4.1.3: 3.00mm Pure Sample FESEM image

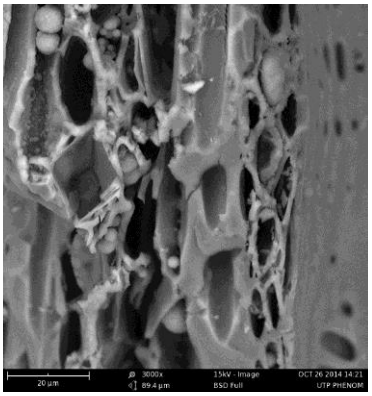


Figure 4.1.4: Sample D1 FESEM image

Based on the FESEM imaging, Figure 4.1.1 and Figure 4.1.2 displays the passage construction in charcoal sample with 0.25 mm size before and after the carbonization process is being carried out at 500°C, which is essential in production of activated carbon. The canal permits the charcoal to absorb the chemical activation agent to activate pore development. Figure 4.1.1 and Figure 4.1.2 is the 3000 times magnification on pure sample and sample A2 respectively. The image clearly shows that pores have already been formed on the sample at 500°C and two hours of carbonization. Bigger pores are observed on the surface morphology of sample A2 as shown in Figure 4.1.2.

Figure 4.1.3 and Figure 4.1.4 displays the passage construction in charcoal sample before and after the carbonization process being carried out in sample sized 3.00 mm at 600°C. Figure 4.1.3 shows the pore formation in good condition but Figure 4.1.4 shows the porous wall are broken slightly at 600°C of carbonization temperature for two hours. It 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 Label	Activation Temperature (°C)	Sample Size (mm)	Impregnation ratio	S_{BET} (m^2/g)	V_T (cm^3/g)	D (nm)
Pure Sample	-	0.25	-	1.4202	0.004343	7.695
A1	500	0.25	1:10	241.5382	0.14126	2.349
A2	**500**	**0.25**	**1:10**	**415.5244**	**0.22812**	**2.347**
B1	500	3.00	1:5	76.9645	0.04694	2.448
E1	500	0.25	1:1	1.4102	0.003759	10.065
E2	600	0.25	1:1	364.8818	0.200986	2.232
F1	500	3.00	1:1	0.0827	-	-
F2	500	3.00	1:3	323.7159	0.15229	9.482
J2	600	0.25	1:3	365.2791	0.196245	2.134

Table 4.3: Surface Area & Porosity Analysis

The table above shows the resulting surface area of the pore formed on activated carbon derived from charcoal at different preparation conditions. Based on the results, it shows that 600°C is the maximum carbonization temperature for charcoal based activated carbon and any higher activation temperature. Earlier results from FESEM showed A2 as having a bigger pore size and larger quantity of Carbon element from the elemental composition result.

Sample A1, B1, E1, and F1 are all carbonized at a 500°C which yields lower S_{BET} which is 241.5382, 76.9645, 1.4102, and 0.0827 respectively at a carbonization temperature of 500°C. While the sample A2, E2, F2 and J2 are all samples that has been carbonized at 600°C which also yields S_{BET} of 415.5244, 364.8818, 323.7159, and

365.2791 respectively at different impregnation ratio. It can be concluded that the samples that have a bigger sample size yields lower BET surface area and lower total pore volume. Sample that has a smaller size which is 0.25mm yields higher BET surface area and higher total pore volume. Among all the samples, A2 yields the higher BET surface area and highest total pore volume.

By comparing the impregnation ratio of the samples, the sample that has a lower impregnation ratio yields lower BET surface area and lower total pore volume while the samples that has higher impregnation ratio has higher BET surface are and higher total pore volume. Therefore, it is suggested that higher impregnation ratio should be used for charcoal to produce activated carbon.

It is also has been proved that all the samples that has been analyzed are at most contains class of mesoporous. The highest pore size obtained was 17.7 nm from sample A2. Even though, the rest of the samples yields pores that has size more than 2nm which is also considered as mesopore but the biggest among all the samples are A2.

It can be concluded; 0.25mm charcoal sample that has been carbonized at 600°C with a higher impregnation ratio yields the best activated carbon product.

4.3 Nitrogen Adsorption-Desorption Isotherm

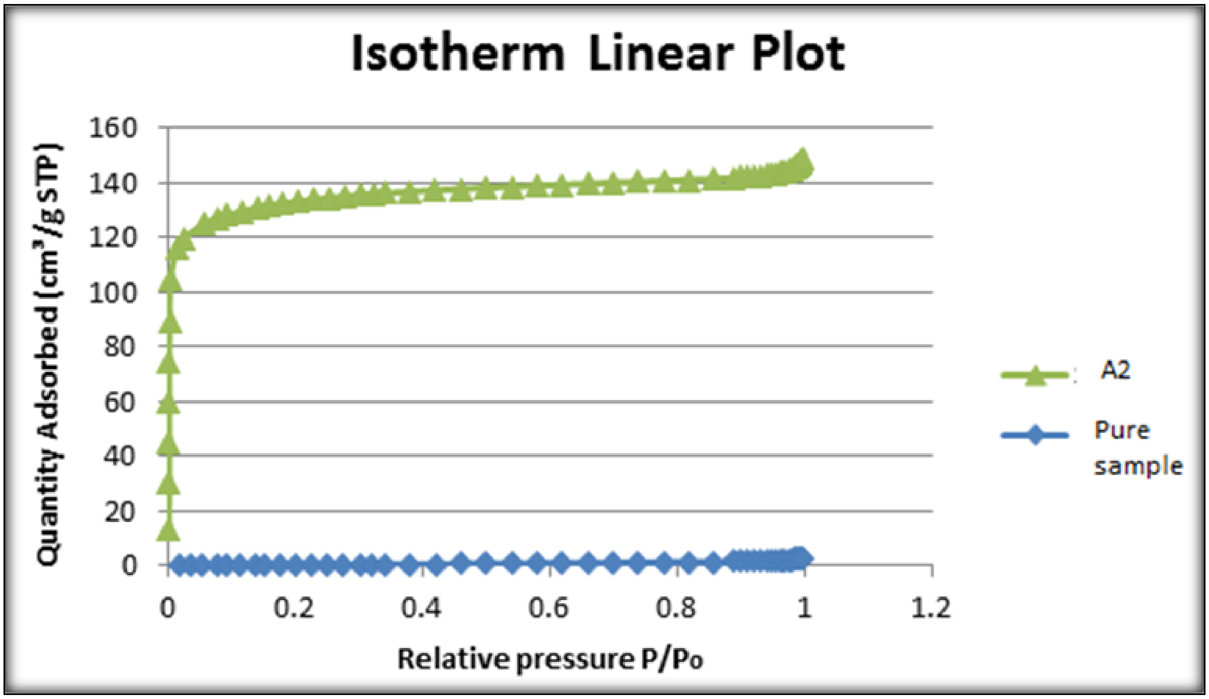


Figure 4.3.1: Nitrogen Adsorption-Desorption Isotherm

Figure 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. 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 4.3.1, pure charcoal sample is identified to follow the isotherm similar to TYPE III where the adsorption is limited to the completion of a single monolayer of adsorbate at the adsorbent surface and it is obtained when the amount of gas adsorbed increases without limit as its relative saturation approaches unity.

Figure above also shows the adsorption-desorption curve for sample A2 which exhibits the characteristics of Type II isotherms which are observed for the adsorption of gases on mesoporous soilds whose pore sizes are not much larger than the molecular diameter of the adsorbate. Complete filling of these narrow pores corresponds to the completion of a molecular monolayer. Adsorption in all other types does not reach a limit corresponding to the completion of a monolayer. This result is evident with BET surface area analysis proving that the pores are in mesoporous group with pore diameter, 17.7 nm.

4.4 Carbon Dioxide Adsorption Analysis

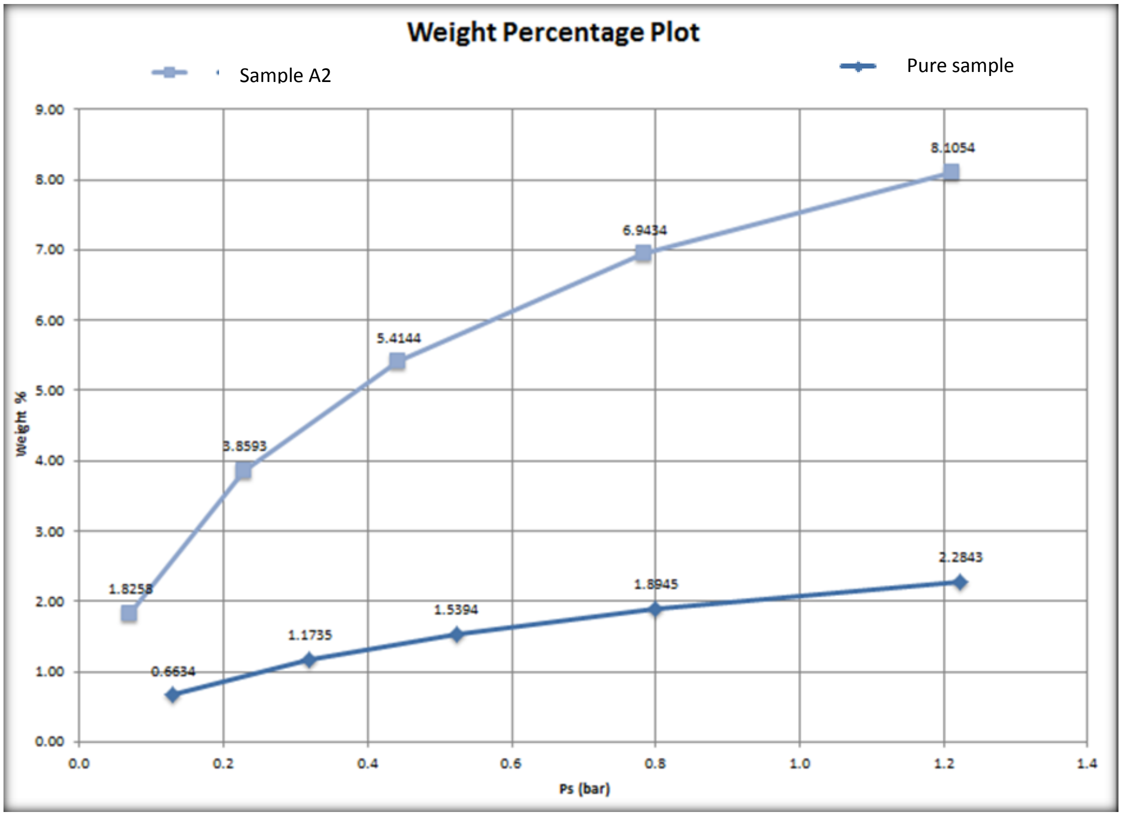


Figure 4.4.1: Weight Percentage of Carbon Dioxide Adsorbed

Figure 4.4.1 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 for both the conventional charcoal (pure sample) and activated carbon (sample A2), proving that activated carbon derived from charcoal is proficient in removing carbon dioxide gas with mesoporous pore formation. The amount of carbon dioxide, CO_2 adsorbed onto the pure sample is 2.2843 weight percentages at standard temperature and pressure, while the amount of carbon dioxide, CO_2 adsorbed onto the sample A2 is 8.1084 weight percentages. The result clearly shows that amount of carbon dioxide adsorbed in the activated carbon produced was much higher than the pure sample. Thus, the activated carbon produced is eligible to adsorb carbon dioxide particles.

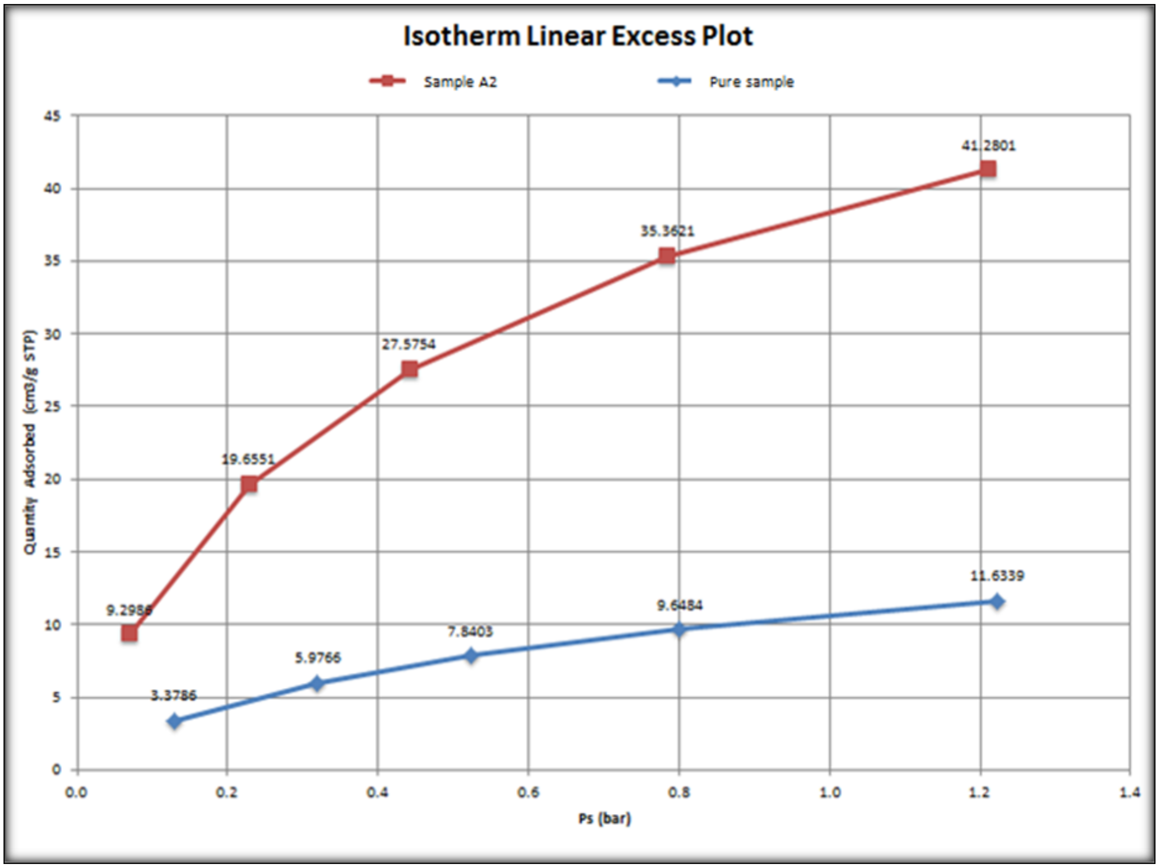


Figure 4.4.2: Adsorption Capacity of Carbon Dioxide on Activated Carbon Derived from Charcoal

The adsorption isotherm in Figure 4.4.2 evidently has the similar shape to the isotherm of weight of carbon dioxide adsorbed. 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 are encountered when adsorption is limited to, at most, only a few molecular layers. This condition is encountered in chemisorption where the asymptotic approach to limiting quantity that all of the surface sites are occupied. In the case of physical adsorption, type I isotherms are encountered with microporous powders whose pore size does not exceed a few adsorbate molecular diameters. A gas molecule, when inside pores of these small dimensions, encounters the overlapping potential from pore walls which enhances the quantity of gas adsorbed at low relative pressures. At higher pressures, the pores are filled by adsorbed or condensed adsorbate leading to the plateu, indicating little or no additional adsorption after the micropores have been filled. Physical adsorption that produces the Type I isotherm indicates that exposed surface resides almost exclusively within the micropores, which once filled with adsorbate, leave little or no external surface for additional adsorption [25].

The effect of temperature on the amount of carbon dioxide, CO_2 adsorbed onto the activated carbon, decreases with increasing temperatures. 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 [26]. Therefore, it is reasonable that adsorption test for carbon dioxide on banana peel based activated carbon is studied at low temperature of 25°C.

5.0 Conclusion & recommendation

5.1 Conclusion

As a conclusion, this undertaking is significant as it manages elective methods for removing carbon dioxide discharges in air. Adsorption utilizing activated carbon is made to be one of the viable approaches to manage the current issue by utilizing conventional charcoal as a part of regular courses particularly in dispensing with carbon dioxide. Consequently, the examination in this task demonstrates that the charcoal is a suitable antecedent to be adjusted into enacted carbon because of its high carbon content. Chemical activation strategy is favored in this study with the utilization of phosphorus acid. In conjunction with that, different readiness parameters have been considered completely to study the impacts on surface area and pore distribution of the activated carbon from charcoal. Along these lines, referring to the results, it is demonstrated that littler molecule size is ideal to be used as artificially initiated carbon as it uncovered bigger surface range among impregnation and actuation. Moreover, the impregnation proportion ought to be genuinely low to hinder great pore development. Besides, the carbonization temperature for utilizing charcoal as an indication should not be higher than 600°c as any higher will realize the breakdown of the permeable arrangement in the enacted carbon and cause the degeneration of powerful surface range for the adsorption process. Test A2 turned out to be the best example among others with its remarkable BET surface region and additionally nitrogen adsorption-desorption isotherm demonstrating meso-porosity. Finally, activated carbon developed from charcoal additionally demonstrated to relinquish carbon dioxide through adsorption.

5.2 Recommendation

The research can be enhanced to the following level, that is, to study the adsorption and also desorption methodology utilizing the charcoal based on activated carbon as adsorbent. As far as adsorption, there are adequate of parameters that can be viewed as, for example, the measure of the adsorbent present, the ph of the arrangement, and the temperature and contact time of the adsorption process. The specimen with the most astonishing S_{BET} from this task (test A2) will be delivered in bulk amount to direct this study. The study on the adsorption methodology is remarkable in deciding the most favorable environment for the carbon dioxide gas to be adsorbed on the reactive areas of the charcoal based on activated carbon which will exceptionally build the adsorption limit of the adsorbent. The time desired to achieve the adsorption balance can be determined through this study. Adsorption equilibrium is attained when the rate of adsorption is equivalent to the rate of desorption, in this way making any further adsorption exceedingly impossible. 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 method.

Also, various sorts of biomass or agroindustry wastes can likewise be inspected on the attainability as precursor to be changed over into activated carbon. Activated carbon produced using various antecedents will have distinctive inclination or adsorption limit, dependent upon its particular planning conditions. Extra data can be acquired through comparative examination with different crude materials. Consequently, the investigation of creating adsorbent developed from rural waste would be an incredible investigation to manage as it gives a finer option monetarily and earth to protect contamination free environment.

6.0 Reference

[1] "Geoengineering the climate: science, governance and uncertainty". The Royal Society. 2009. Retrieved 2011-09-10. Accessed from: https://royalsociety.org/~/media/Royal_Society_Content/policy/publications/2009/8693.pdf

[2] A.Mohammas-Khah and R.Ansari. (Oct-Dec 2009). Activated Charcoal: Preparation, characterization and Applications: A review article, International Journal of ChemTech Research, Vol. 1, No. 4, pp 859-864.

[3] J.Romanos et al. (2012). Nanospace engineering of KOH activated carbon. Nanotechnology, Vol 23, No 1, Accessed 2014-6-21, Retrieved from: http://iopscience.iop.org/0957-4484/23/1/015401/pdf/0957-4484_23_1_015401.pdf

[4] National Oceanic & Atmospheric Administration (NOAA) – Earth System Research Laboratory (ESRL), Accessed 214-06-21. Retrieved from: http://www.esrl.noaa.gov/gmd/

[5] Obersteiner, M., Azar, C., Kauppi, P., Möllersten, K., Moreira, J., Nilsson, S., Read, P., Riahi, K., Schlamadinger, B., Yamagata, Y., Yan, J., and van Ypersele, J. P.: (2001) "Managing climate risk"

[6] Properties of Activated Carbon. CPL Caron Link. Accessed 2014-6-21. Retrieved from http://www.activated-carbon.com/

[7] Simple technologies for charcoal making, (2013). Food and Agriculture Organization of the United Station, Forestry Department. Retrieved from: http://www.fao.org/docrep/X5328e/x5328e02.htm#1.4.1. what is charcoal

[8] M Kato, D M DeMarini, A B Carvalho, M A V Rego, A V Andrade, A S V Bonfim,D Loomis. World at work: Charcoal producing industries in northeastern Brazil. Occupational Environmental Medicine 2005. Volume 62. Pp 128-132.

[9] M. Soleimani, T. Kaghazchi. Agricultural Waste Conversion to Activated Carbon by Chemical Activation with Phosphoric Acid (24 APR 2007). Chemical Engineering & Technology. Volume 30. Issue 39. Pp 649-654

[10] What is activated charcoal and why is it used in filters?. (2000, April). HowStuffWorks.com. [Online]. Available: http://science.howstuffworks.com/environmental/energy/question209.htm

[11] Dr. D. Uhríková, CSc. Adsorption. Accessed 2014-06-22. Retrieved from: https://www.fpharm.uniba.sk/fileadmin/user_upload/english/Physical_Chemistry/5-Adsorption.pdf

[12] Glossary. The Brownfields and Land Revitalization Technology Support Center. Retrieved 2014-06-21. Accessed from: http://www.brownfieldstsc.org/glossary.cfm?lett=A

[13] N.A.Rashidi, S.Yusup, and A.Borhan. (2014). Novel low-cost activated carbon from coconut shell and its adsorptive characteristics for carbon dioxide. UTP Institutional Repository. [Online]. 594-595, pp. 240-244. Retrieved from: http://eprints.utp.edu.my/10637/1/KEM.594-595.240.pdf

[14] C.Cui-Xia, H.Biao, L.Tao, W.Geng-Feng, Preparation of phosphoric acid activated carbon from sugarcane bagasse by mechanochemical processing. Bioresources.com. Accessed 2014-06-22, Retrieved from : http://ojs.cnr.ncsu.edu/index.php/BioRes/article/view/BioRes_07_4_5109_Chen_Phosphoric_Acid_Sugarcane_Bagasse_Mechanochemical/1751

[15] S.M. Yakout, G. Sharaf El-Deen (2012). Characterization of activated carbon prepared by phosphoric acid activation of olive stones, Arabian Journal of Chemistry.

[16] The Naked Whiz's (2001). Accessed 2014-06-22. Retrieved from: http://www.nakedwhiz.com/lumpfaq.htm

[17] Happi Emaga, T. ; Bindelle, J. ; Agneesens, R. ; Buldgen, A. ; Wathelet, B. ; Paquot, M., 2011. Ripening influences banana and plantain peels composition and energy content. Trop. Anim. Health Prod., 43 (1): 171-177

[18] Shulga (2007). Lignocellulosic mulch, polycomplex, soil. BioResources 2(4), pg 572-582.

[19] K. mojsov, M. Petreska. J.Ziberoski. Biosynthesis of Pectinolytic enzymes by Aspergillus on apple pulp.

[20] Krishnan, K.A., "Adsorption of nitrilotriacetic acid onto activated carbon prepared by steam pyrolysis of sawdust: Kinetic and isotherm studies", Colloids and Surfaces A: Physicochemical and Engineering Aspects, 317 (2008), 344-351.

[21] Wu, F.C., Tseng, R.L., Juang, R.S., "Comparisons of porous and adsorption properties of carbons activated by steam and KOH", Journal of Colloid and Interface Science 283 (2005) 49–56.

[22] A.Borhan, A.F. Kamil. Preparation and Characterization of Activated Carbon from Rubber-seed shell by Chemical Activation (2012). Journal of Applied Sciences 12.

[23] A.C Lua. T.Yang. Characteristics of activated carbon prepared from pistachio-nut shell by zinc chloride activation under nitrogen and vacuum conditions (2005).

[24] M. K. Lam, Z. Riduan. Production of activated carbon from sawdust using fluidized bed reactor. International Conference on Environment (ICENV 2008).

[25] GAPP Applications(2014). Accessed 2014-22-12, Retrieved from http://www.app-one.com.cn/eng/newsInfo.aspx?parentid=940&catid=944&newsid=11956

[26] Heidari.A., Younesi.H., Rashidi.A., Ghoreyshi.A., 2013, "Adsorptive removal of CO_2 on highly microporous activated carbons prepared from *Eucalyptus camaldulensis* wood: Effect of chemical activation", Journal of the Taiwan Institute of Chemical Engineers, **45:** 579 – 588.

7.0 Appendices

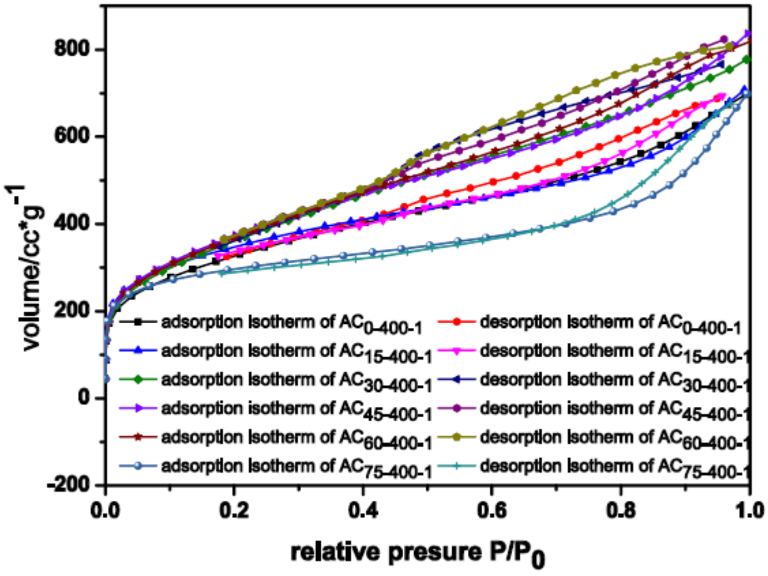


Figure 7.0.1 : Adsorption-desorption isotherm of N_2

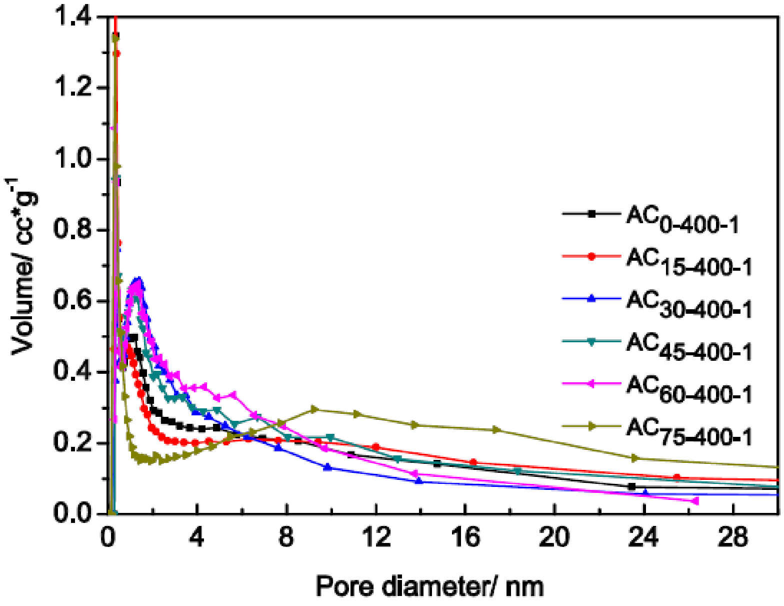


Figure 7.0.2 : Pore size distribution of activated carbon

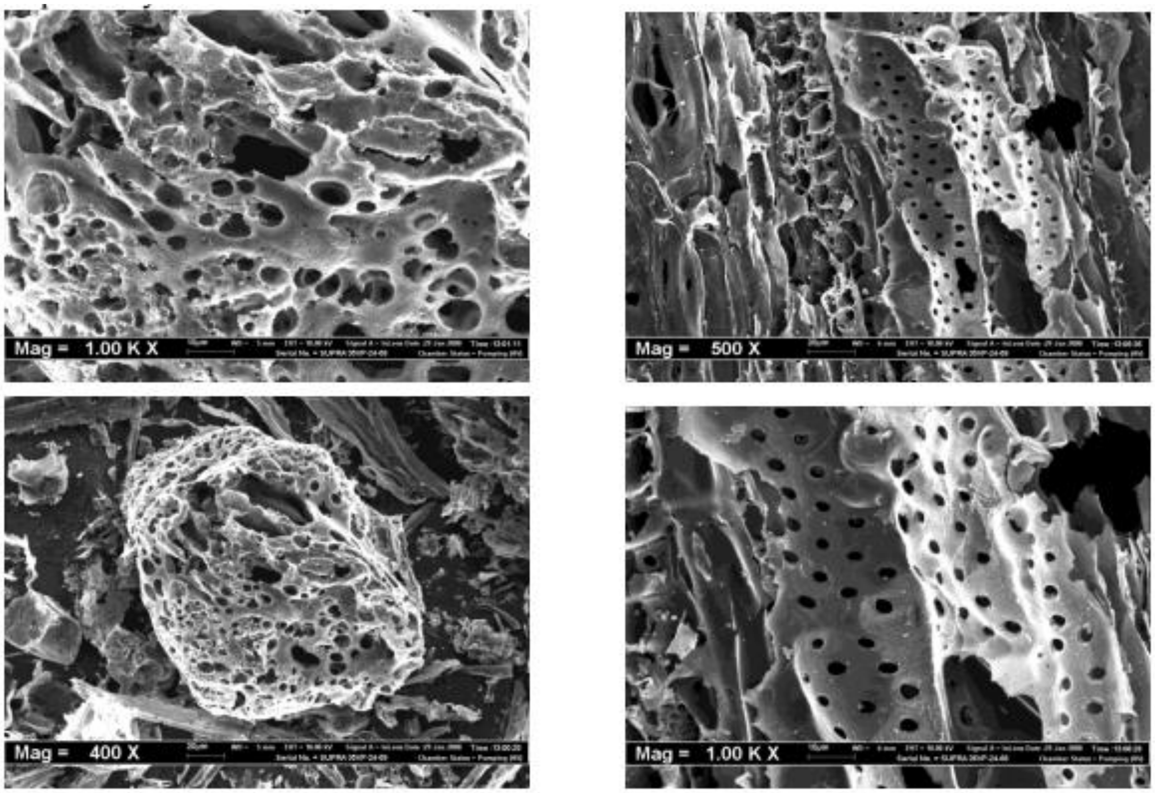


Figure 3 (a) and (b) SEM of AC0.5-500-2

Figure 4 (a) and (b) SEM AC1-500-2

Figure 7.0.3: Scanning Electron Microscope(SEM) studies

The surface morphologies of activated carbon produced was determined using SEM. Figure 3(a) and (b) shows the SEM photograph of AC0.5-500-2 at 400x and 1000x magnifications, respectively. Figure 4(a) and (b) shows the SEM photograph of AC1.0-500-2 at 500x and 1000x magnifications, respectively. It is expected to obtain a result similar like this image for this project as well [24].

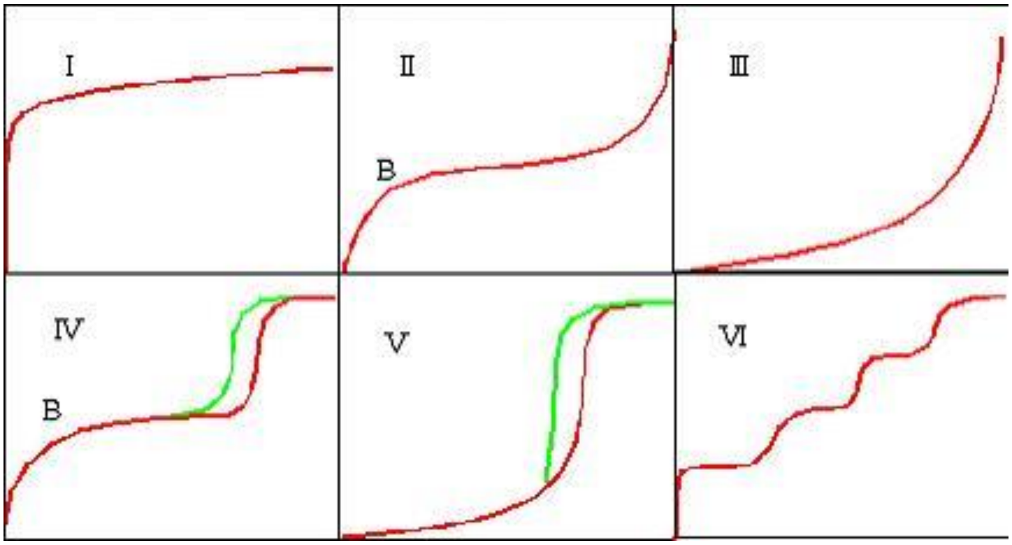


Figure 7.0.4: Types of isotherms

BET Theory put forward by Brunauer, Emmett and Teller explained that multilayer formation is the true picture of physical Adsorption. One of the basic assumptions of Langmuir Adsorption Isotherm was that adsorption is monolayer in nature. Langmuir adsorption equation is applicable under the conditions of low pressure.