SYNTHESIS AND CHARACTERIZATION OF ADSORBENT FOR HYDROGEN STORAGE

By

TAN SOOK YEE

FINAL PROJECT REPORT

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the requirements for the
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Universiti Teknologi Petronas
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved:

A.P. Dr. Suzana Yusup
Research Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

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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.

Tan Sook Yee

ABSTRACT

Hydrogen is current potential for new energy applications. Thus, Methods of hydrogen storage have been widely discussed for the advancement of hydrogen and fuel cell power technologies since the problem of hydrogen storage has become the bottleneck for adoption of hydrogen as energy carrier. The objective of this research is to record the detailed experiment procedure to synthesize and characterize the adsorbent developed from sugarcane bagasse. The scope of project work includes the synthesis of adsorbent produced from environmentally friendly agriculture waste – sugarcane bagasse which consists of high cellulose content, and followed by the characterization of the new series of chemically activated carbon. Important findings and discussion of the research work are also discussed. Activating agent of $ZnCl_2$, KOH and NaOH solutions with different concentration were used for chemical activation before undergoing the carbonaceous stage. The sample is washed with distilled water before characterization is performed. Effect of type of activating agent and its concentration on the porosity development were discussed. Characterization criteria of porosity, pore size distribution, surface roughness using SEM and carbon content using EDX analysis were discussed in Chapter 4. The SEM images show that the smooth, spongy and uniform pore structure is obtained by activation of KOH and NaOH solutions if compared with $ZnCl_2$ activated carbon. Other criteria such as specific surface area, micropore surface area, pore volume, density, and pyrolysis condition that will affect the hydrogen adsorption were also discussed in literature review and discussion.

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CHAPTER 1
INTRODUCTION

1.1 Background

Hydrogen energy may on its way to becoming major, environmentally benign, sustainable, renewable component of the world's energy mix for both transportation and stationary applications. As an idea from Peter H. et al., hydrogen – an invisible, tasteless, colorless gas – is the most abundant element in the universe. Highly reactive, it is essential in innumerable chemical and biological processes. It is an energetic yet non polluting fuel. When burned in an internal-combustion engine (piston, rotary, or gas turbine), hydrogen produces as exhaust virtually nothing but harmless water vapor (plus, admittedly, trace emissions from tiny amounts of engine lubricants that are oxidized in the process, and some nitrogen oxides). When hydrogen is combusted with atmospheric oxygen in an engine, no carbon monoxide or carbon dioxide is emitted, no unburned hydrocarbons, no stench, no smoke, nor any of the other carbon-bearing, Earth-befouling discharges we suffer today [Peter H. et al. 2002].

Among all the chemicals, hydrogen exhibits the highest heating value per mass as shown in Table 1, which means hydrogen would release the greatest amount of heat during combustion of a specified amount of it compared with other common fuels.

Table 1.1: Heat of combustion for common fuels [wikipedia.org]

	Heat of combustion		
[illegible]	kJ/g	kcal/g	BTU/lb
Hydrogen	141.9	33.9	61,000
Gasoline	47	11.3	20,400
Diesel	45	10.7	19,300
Ethanol	29.8	7.1	12,800
Propane	49.9	11.9	21,500
Butane	49.2	11.8	21,200
Wood	15	3.6	6,500
Coal	15-27	4.4-7.8	8,000-14,000
Natural Gas	~54	~13	~23,000

1.2 Problem Statement

Pure hydrogen gas does not exist as a natural resource like oil. It must be extracted from some other natural resources like water, methane, biomass, coal or oil [The Hydrogen Economic, www.americanenergyindependence.com]. Thus, methods of hydrogen storage have been widely discussed for the advancement of hydrogen and fuel cell power technologies. Based on the storage technologies investigated in this few years, hydrogen can be stored in compressed tanks, in liquefied form, metal hydrides and adsorbent packed bed [Hydrogen Storage, www.rsc.org]. In case of metals and inter-metallic compounds, the negative factors for hydrogen storage are either the limitation of storage capacity or the reversibility of stored hydrogen under favor experimental condition [J. S. Im, et al. 2008]. Filling a fuel tank with hydrogen is not as easy as filling a tank with gasoline. The energy density by volume of liquid hydrocarbons such as gasoline or diesel is much greater than the energy density of either compressed or liquid hydrogen [The Hydrogen Economic, www.americanenergyindependence.com]. As written by Joseph M. N. et al, storage of hydrogen as a cryogenic liquid is currently the large-scale method being employed by industry. However, the cost of liquid hydrogen is extremely high due to the liquefaction process. The problem of hydrogen storage efficiently has become one of the major technological barriers currently preventing the widespread adoption of hydrogen as an energy carrier and it is believed to be one of the most difficult challenges facing the hydrogen economy.

Hydrogen gas has good energy density by weight, but poor energy density by volume versus hydrocarbons and hence it requires a larger tank to store [The Hydrogen Economic, www.americanenergyindependence.com]. In comparison with a tank of hydrocarbon, a much larger tank is needed in order to store the same amount of energy. Gaseous hydrogen is conventionally stored in banks of "K" bottles or "tube" tanks at pressure as high as 6000psig [Joseph M. N., et al.]. Increase in gas pressure would improve the energy density by volume, thus, smaller tank required but heavier in result. In addition, large external power is required in order to compress the gaseous hydrogen which will cause more in energy lost and thus, it is not preferable as hydrogen storage method.

Liquid hydrogen having the higher energy density may be used alternatively. But one of the problems with using liquid hydrogen is that the liquefaction process is energy intensive and involves several steps, including (1.) the compression of gaseous hydrogen with reciprocating compressors; (2.) the precooling of the compressed gas to liquid nitrogen temperature (78K); followed by (3.) expansion through turbines [Joseph M. N., et al.]. Such process may impose to higher cost than gaseous hydrogen and large energy lost during the cooling stage and thus, tank must be well insulated to prevent boil off. Ice may form around the tank and corrodes the tank if the insulation fails [Hydrogen Storage, www.rsc.org].

1.3 Objective and Scope of Study

The main objective of the research is to prepare a new series of chemically activated carbon through improvement of the developed synthesis approach and analyze their usage for hydrogen storage. The scope of study will consist of:

- Synthesize agriculture waste to be improved as adsorbent sample through a well developed methodology, by manipulating the activating agent and its concentration.
- Perform characterization on the new prepared adsorbent. Study of adsorption isotherm, particle size, pore size distribution, surface roughness, pore volume and average pore diameter will be included.
- Analyze on the hydrogen adsorption capacity of the developed adsorbent for hydrogen storage.

CHAPTER 2

LITERATURE REVIEW

2.1 Adsorbent for Hydrogen Storage

Adsorption is the binding of molecules or particles (adsorbate) to a solid surface (adsorbent) due to the unsaturated and unbalanced molecular forces that are present on every solid surface. The adsorption involves two types of forces: physical forces that may be dipole moments, polarization forces, dispersive forces, or short-range repulsive interactions and chemical forces that are valency forces arising out of the redistribution of electrons between the solid surface and the adsorbed atoms. Activated carbons are versatile adsorbents. Their adsorptive properties are due to their high surface area, a microporous structure, and a high degree of surface reactivity. [R. C. Bansal, et al. 2005]

2.2 Earlier Research Done

There are some researches works that had been done earlier regarding the development of adsorbent for hydrogen storage. The following will summarize the research work done by earlier researchers with the methodology and important findings.

Table 2.1: Research works done by earlier researchers.

Research Title	Researcher	Year
Hydrogen Storage by Carbon Materials Synthesized from Oil Seeds and Fibrous Plant Materials	Maheshwar Sharon, T.Soga, Rakesh Afre, D.Sathiyamoorthy, K.Dasgupta, Sunil Bhardwaj, Madhuri Sharon, Sandesh Jaybhaye (India and Japan)	2007
Production of Activated Carbon from Rubber Wood Saw Dust	C.Srinivasakannan, Mohamad Zailani Abu Bakar (School of Chemical Engineering, Engineering Campus, University Sains Malaysia, Malaysia)	2004
Hydrogen Adsorption Characteristic of Activated Carbon	Hangkyo Jin,Young Seak Lee, Ikpyo Hong (Republic of Korea)	2007
Mesopore Control of High Surface Area NaOH Activated Carbon	Ru Ling Tseng (Department of Safety, Health and Environmental Engineering, National United University, Taiwan)	2006
The Study of Controlling Pore Size on Electrospun Carbon Nanofibers for Hydrogen Adsorption	Ji Sun Im, Soo-Jin Park, Tae Jin Kim, Young Ho Kim, Young-Saek Lee (Republic of Korea)	2008
Hydrogen Adsorption by Nanostructured Carbons Synthesized by Chemical Activation	M.Zyzlila Figueroa-Torres, Alejandro Robau-Sanchez, Luis De la Torre-Saenz, Alfredo Aguilar-Elguezabal (Chihuahua, Mexico)	2007

2.2.1 Hydrogen Storage by Carbon Materials Synthesized from Oil Seeds and Fibrous Plant Materials

The material used in the research is the series of plant based fiber (bagasse, corn, rice straw, jute straw, coconut fiber, bamboo) and plant based seeds. Samples are treated with aqueous solution of HCl and heat treated under vacuum condition. The dried samples were carbonized in the furnace with the pyrolysis temperature constantly at 850°C and soaking time range of 3 to 4 hours. Both nitrogen and hydrogen gas flow was studied, with the flow rate set constantly at 6ml/min. After cooling, the carbon materials were taken out and powdered. Since soybeans and bagasse show the highest hydrogen adsorption, these two precursors were further studied by varying the temperature (600-800°C). After carbonization, chemical activation were done by soaking the char in HCl and KOH for 1 hour and washed with water and acetone. The materials were dried at 100°C.

As a result, the densities of seed based carbon are greater than densities of plant based carbon, range of 0.5 to 0.9 g/cc and 0.2 to 0.5 g/cc respectively. The yield is in a range of 20% to 31%. The surface area of plant based carbon is greater than seed based carbon, with the range of 61.29 to 187.66 m^2/g and 12.83 to 67.7 m^2/g respectively. Hydrogen adsorption of plant based carbon is in the range of 0.08 to 0.66 wt%, which is greater than hydrogen adsorption of seed based carbon, in the range of 0.07 to 0.23 wt%. Bagasse under pyrolysis temperature of 700°C with hydrogen gas flow and 3M KOH treatment shows the highest hydrogen adsorption of 2.67 wt%. While the soybean under pyrolysis temperature of 700°C with argon gas flow and no chemical treatment shows the second highest hydrogen adsorption of 2.00 wt%. As conclusion, the carbon materials from plant based give better hydrogen adsorption. Lower density and larger surface area are obtained from fibrous precursors, while the bagasse shows agglomerated tubular fibers forming channel type structure.

2.2.2 Production of Activated Carbon from Rubber Wood Saw Dust

Rubber wood sawdust was used for analysis in this research. The pre-dried sawdust was mixed with the phosphorus acid. The impregnation was performed overnight and semi-carbonized at 200°C for 15 minutes. After the furnace reaches the desired activation temperature (400°C and 500°C), the sample was then sent into furnace for activation. The activation time is range from 30 to 90 minutes. Washing was done after activation until the wash liquor become neutral. The product was sieved at 300 µm.

In the findings, the highest BET surface area obtained is 1492 m^2/g, at 500°C for 45 minutes activation time. The product yield is range from 33 to 63%. Low activation time and temperature result in incomplete burn off resulting in a higher yield. Thus, the optimum temperature is at 500°C. Beyond the optimum temperature and time, reduction in surface area is observed, which can be attributed to the increase in pore size leading to collapse of the small pores. A similar trend in the increase of surface area in the impregnation ratio until an optimum value and reduction with further increase in the impregnation ratio is also reported in the rest of researches.

2.2.3 Hydrogen Adsorption Characteristic of Activated Carbon

Coconut shell was used as the material in the analysis. The coconut shell was crushed up in a particle size smaller than 0.2 mm. Powder raw material was then mixed with KOH in vibration ball mill. The mixing ratio (KOH/coconut shell) ranges from 1:1 to 1:5 in weight was prepared. The mixture was heated in a tubular furnace at 5K/min up to the activation temperature of 700°C, 800°C, and 900°C in argon inert atmosphere. Activation was performed for 2 hours. The product was then washed with distilled water and dried at 150°C for 3 hours.

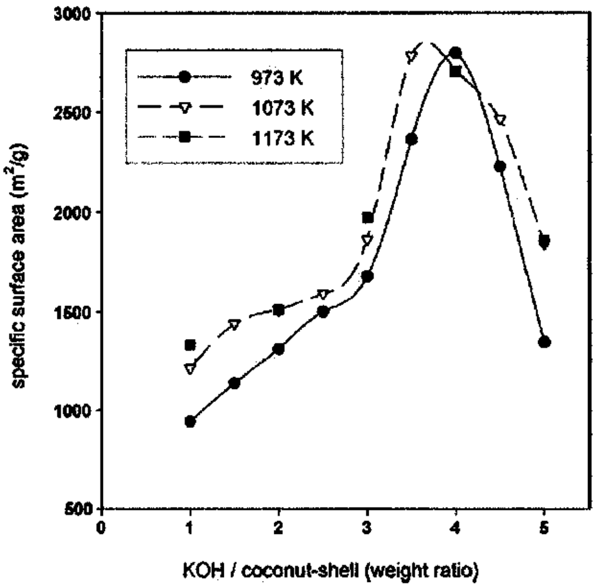

Figure 2.1: Effect of KOH concentration on the micropore surface area of activated carbon prepared at different activation temperature.

Refer to the Figure 2.1, the specific area is found increased according to KOH/coconut shell ratio up to 3.5 to 4.0, then decreased regardless of activation temperature. Maximum specific surface area reached up to 2800 m^2/g is obtained. Increase the temperature from 700°C to 800°C increased the specific area, but no difference in specific surface area between 800°C and 900°C. The micropore area increased according to KOH/coconut shell ratio up to 3.5 to 4.0, and then decreased in similar manner regardless of activation temperature. The total pore volume increased according to KOH/coconut shell ratio up to 4.0 to 4.5, and then decreased regardless of activation temperature. The hydrogen adsorption capacity increases almost linearly according to their specific surface area. Maximum hydrogen capacity is 0.85 wt% with 0.06 pore filling degree. Total pore range from 0.43 to 2.17 cm^2/g. Hydrogen adsorption capacity range from 0.44 to 0.85 wt% while the degree of pore filling range from 0.05 to 0.16. Degree of pore filling can be calculated by dividing the measured H_2 adsorption capacity by theoretical H_2 adsorption capacity. The theoretical H_2 adsorption capacity can be calculated by multiplying total pore volume by liquid hydrogen density, which is 0.07 cm^3/g.

As conclusion, the hydrogen adsorption capacities depend almost linearly on porous structure parameters: specific surface are, micropore surface area, total pore volume and micropore volume. The well-developed microporous material seems to be the promising candidate for hydrogen storage.

2.2.4 Mesopore Control of High Surface Area NaOH Activated Carbon

A study of relationships between pore properties and NaOH dosage had been carried out by the author. Comparison between the methods of NaOH and KOH activation revealed that NaOH activation can suitably control the mesopore specific volume of the activated carbon. The material used in this research is corncob. The corncob was carbonized into char for 1.5 hours, and then soaked in a concentrated NaOH solution, oven dried before activated. The char sieved to a uniform size ranging from 0.833 to 1.65 mm. The char is then activated at 780°C for 1 hour. The samples were classified according to the NaOH/char weight ratio, according to 0.5 to 6, with a step size of 0.5.

The average pore diameter for NaOH/char ratio of 0.5 to 3.0 is 3.0 nm, while the average pore diameter for NaOH/char ratio of 4.0 to 6.0 is 4.2 nm. The optimum agent or char ratio to produce highest specific surface area at 2500 m^2/g is 4.0. Highest pore volume of about 1.7 cm^2/g is obtained at the ratio of 6.0. The elemental analysis on the activated carbon shows that they contain C, H, N, O and S. Contracted, twisted, honeycomb shaped holes is observed from the SEM images. The carbon content is in the range from 74.02 to 80.16 wt%. This research concludes that with different ratio of agent/char, the activation divided into micropore development stage and pore volume expanding stage.

2.2.5 The Study of Controlling Pore Size on Electrospun Carbon Nanofibers for Hydrogen Adsorption

Polyacrylonitrile (PAN)-based carbon nanofibers (CNFs) were prepared using electrospinning method and heat treatment to get the media for hydrogen adsorption storage. The electrospinning method is a very simple and efficient way for the fabrication of nano- to microscale fibers which has much smaller diameter than that of conventional carbon fibers by applying electrical potential between a droplet of polymer solution or melt held at the end of a capillary and a grounded collector. PAN and DMF (N,N-dimethyl formamide) were used to prepare the polymer solution. $ZnCl_2$ was used to prepare the polymer solutions with various weight ratios of PAN:DMF:$ZnCl_2$ = 3:30:0, 3:40:2, 3:50:4, 3:60:6. Each polymer solution was heated at 110°C for 1 hour. Oxidation of the electrospun fibers was carried out by heating to 250°C for 8 hours under natural flow of air, and then carbonized at 1050°C for 1 hour in nitrogen atmosphere. On the other hand, PAN and DMF sample was immersed in each KOH solution with 4, 6, and 8M respectively for over night and activated in furnace at 750°C for 3 hours in nitrogen atmosphere. KOH activated CNFs were washed with distilled water and dried over night at 110°C.

In the findings, the surface roughness of KOH activated CNFs is smooth and the diameter is uniform in the same condition of activation. The diameter of KOH activated CNFs decreased with increasing KOH solution mole. It is supposed due to the effect of oxygen in the presence of KOH solutions that would burn off the outside of CNFs. Figure 2.2 shows the effect of oxygen on carbon yield during KOH activation.

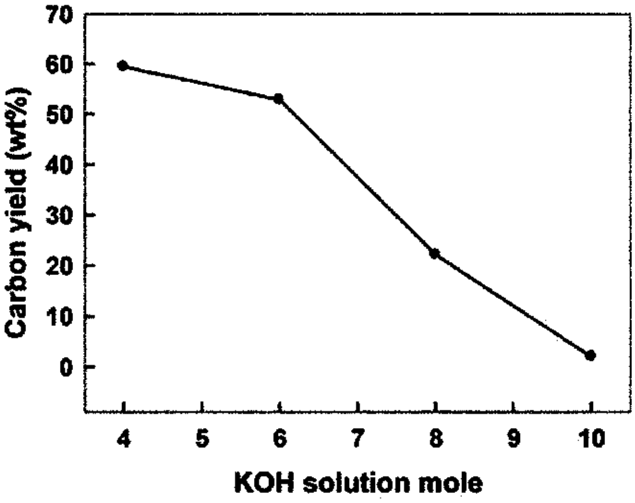

Figure 2.2: Carbon yield of KOH activated CNFs.

The specific surface area of KOH activated CNFs is greater than $ZnCl_2$ activated CNFs, with the range of 1410 to 2420 m^2/g and 334 to 944 m^2/g respectively. As well as for total pore volume where the total pore volume for KOH activated CNFs is range from 0.75 to 2.39 cm^3/g and 0.43 to 0.96 cm^3/g for $ZnCl_2$ activated CNFs. As for hydrogen adsorption, KOH activated CNFs result in range of 0.67 to 1.03 wt%, while $ZnCl_2$ activated CNFs result in range of 0.53 to 1.54 wt%. From the experiment, KOH activation is much more effective to increase specific surface area and total pore volume than $ZnCl_2$ activation. However, in case of getting pore volume in the range of pore width from 0.6 to 0.7 nm, $ZnCl_2$ activation is more effective than KOH activation. The diameter of dynamic hydrogen molecule is 0.4059 nm, thus the most effective factor to attract hydrogen adsorption capacity positively is the pore volume in the range of pore width mainly from 0.6 to 0.7 nm.

2.2.6 Hydrogen Adsorption by Nanostructured Carbons Synthesized by Chemical Activation

In this research, activated carbons were produced by Quercus agrifolia (Coast Live Oak) with chemical activation using NaOH and KOH. NaOH and KOH were ground to powder and mixed physically with char at a given proportion of 4/1. Activation temperature of 760°C and four different nitrogen flow rates of 150, 250, 375, and 500 cm^3/min were used.

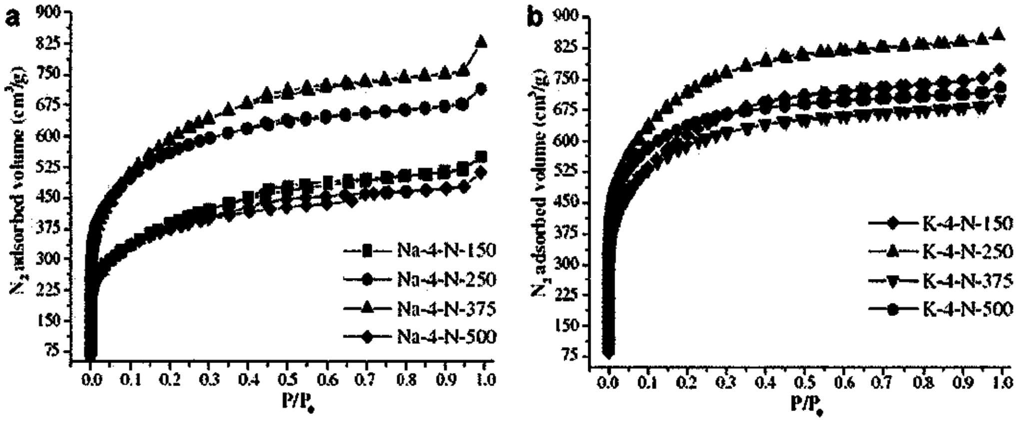

Figure 2.3: N_2 adsorption isotherm at 77K for the activated carbons using (a) NaOH and (b) KOH as activation agents.

As for result, the specific surface area of KOH activated carbon at highest value of 3081 m^2/g with N_2 flow of 250 cm^3/min shows greater than NaOH activated carbon which gives the highest specific surface area of 2252 m^2/g with N_2 flow of 375 cm^3/min. As shown in Figure 2.3, the obtained isotherms are corresponding to Type I, characteristic of microporous materials. However, NaOH activated carbon contributed more to mesoporosity. The average micropore width for all samples, according to Horvath-Kawazoe equation, showed values of 5.5 Å. The contribution of microporosity to total pore volume is higher than the contribution of mesoporosity. In the study of hydrogen adsorption, KOH activated carbon with optimum N_2 flow of 150 and 500 cm^3/min gives 2.71 wt% of hydrogen adsorption, while NaOH activated carbon with optimum N_2 flow of 375 cm^3/min gives hydrogen adsorption of 2.42 wt%. The isotherms are formed by the hydrogen adsorption like a monolayer, i.e., most hydrogen molecules are directly attached to the surface, and a few hydrogen molecules are deposited on the hydrogen previously attached to the surface, since in all cases the amount of adsorbed hydrogen measured is lower than that required to cover 25% of the availability surface. As shown in Figure 2.4, the isotherm pattern for each NaOH sample is different. However, the KOH samples show a very similar isotherm pattern.

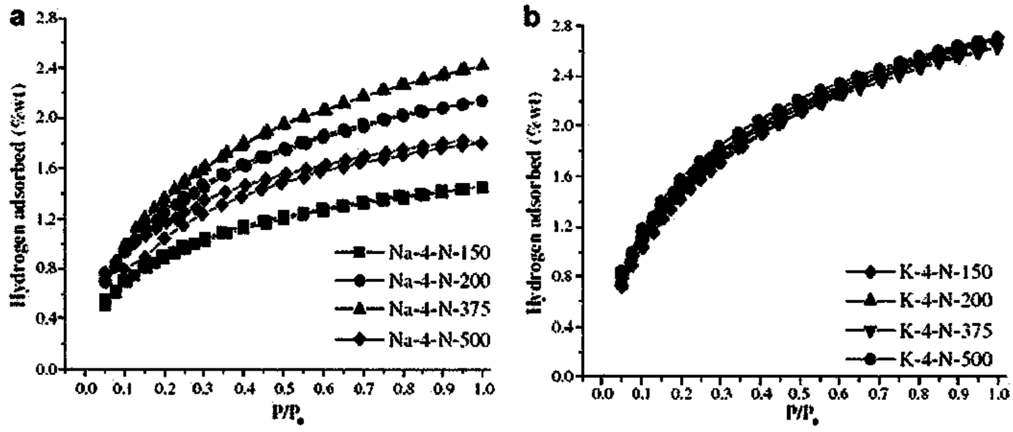

Figure 2.4: Hydrogen adsorption isotherm at 77K using (a) NaOH and (b) KOH as activation agents.

According to the results, the increasing of N_2 flow during the activation process enhances the removal of reaction products, shifting the activation reaction toward the right. For hydrogen adsorption capacity, there is no influence of N_2 flow factor for the case of KOH activation, suggesting that the pore formation could be related to a phenomenon no dependent on the reaction course, i.e. the diffusion of metallic potassium into the carbon matrix. The metallic potassium would separate the atomic layers of carbon and thus produce homogenous nanostructured porosity.

2.3 Material Used

Carbon-based absorbents are well known as one of the better absorbent groups for their ability to exist in a very fine powdered form with a highly porous structure. One viable option for activated carbon is sugarcane bagasse. Activated carbon produced by chemical activation of sugarcane bagasse is proposed and KOH, NaOH, and $ZnCl_2$ will be used for the activation process.

Sugarcanes were widely used in production of sugar, sweet syrup, molasses, rum, soda, and ethanol for fuel. The bagasse that remains after sugarcane crushing may be burned to provide both heat that used in the mill and electricity [Bagasse, wikipedia.org]. It may also, because of its high cellulose content, be used as raw material for paper and cardboard, branded as "environmentally friendly", as it is made from a by-product of sugar production [Sugarcane, wikipedia.org]. Thus, an idea has come to mind that uses the sugarcane bagasse which contain fibrous residue to prepare the new series of chemically activated carbon for hydrogen storage.

Sugarcane is common in tropical and subtropical countries throughout the world, thus low cost. Brazil is the largest sugarcane producer producing about 514 million tons of sugarcane, followed by India with sugarcane production of about 355 million tons. According to the source of Food and Agricultural Organization of United Nations, about 195 countries produce about 1,557 million tons of sugarcane as updated on 11th June 2008 [Sugarcane, wikipedia.org]. The sugar industry in Malaysia is characterized by rapidly increasing direct domestic consumption supported by an equally fast growing food processing industry, and on the supply side by a small domestic production base that is unlikely to expand. To meet the growth in demand, imports have expanded steadily to record levels in recent years. With excess refining capacity, some of the imports of raw cane sugar are refined and re-exported to regional markets [Proceedings of the Fiji/FAO 1997 Asia Pacific Sugar Conference, www.fao.org]. A sugar factory produces nearly 30% of bagasse out of its total crushing [Bagasse, wikipedia.org]. Thus, sugarcane bagasse is easily obtained.

2.4 Adsorbent Characterization

Activated carbon in its broadest sense includes a wide range processed amorphous carbon-based materials. The storage capacity is depends on the adsorbent characteristic and the operational conditions [M. Sharon, et al. 2007]. Activated carbons are high in porosity, high surface area materials manufactured by carbonization and activation of carbonaceous materials, hence high surface capacity for adsorption [C. Srinivasakannan, et al. 2004]. The classification of pores suggested by Dubinin and accepted by the International Union of Pure and Applied Chemistry (IUPAC) is based on their width, which represents the distance between the walls of a slit-shaped pore or the radius of a cylindrical pore. The pores in activated carbons are divided into three groups: the micropores with diameters less than 2 nm, mesopores with diameters between 2 and 50 nm, and macropores with diameters greater than 50 nm. The micropores constitute a large surface area (about 95% of the total surface area of the activated carbon) and micropore volume and, therefore, determine to a considerable extent the adsorption capacity of a given activated carbon, provided however that the molecular dimensions of the adsorbate are not too large to enter the micropores [R.C. Bansal, et al. 2005].

Besides the crystalline and porous structure, an activated carbon surface has a chemical structure. The adsorption capacity of an activated carbon is determined by the physical or porous structure but strongly influenced by the chemical structure of the carbon surface. In graphites that have a highly ordered crystalline structure, the adsorption capacity is determined mainly by the dispersion component of the van der Walls forces. But the random ordering of the aromatic sheets in activated carbons have limited dimensions and therefore constitute edges, in addition of association with defects, dislocations, and discontinuities, causes a variation in the arrangement of electron clouds in the carbon skeleton and result in the creation of unpaired electrons and incompletely saturated valencies, and are richer in potential energy. Consequently, these carbon atoms are highly reactive and constitute active sites or active centers, which would undoubtedly influence the adsorption properties of activated carbons [R.C. Bansal, et al. 2005].

2.4.1 Scanning Electron Microscope (SEM)

The surface morphology and textural properties of the developed adsorbent will be investigated using SEM to observe the pore size change through activation, pore diameter and surface roughness. Since the hydrogen adsorption capacities of activated carbon can be determined in relation with their textural characteristics [N. Texier-Mandoki, et al. 2004].

2.4.2 Brunauer, Emmett and Teller (BET) Theory

The formation of multimolecular layers was explained by assuming that the uppermost layer of the adsorbent induces dipoles in the first layer of adsorbate molecules, which in turn induces dipoles in the second layer, and so on until several layers are built up. Brunauer, Emmett, and Teller were of the view that the polarization of the second layer of adsorbed gas molecules by the first layer gas molecules will be much too small to constitute a major portion of the binding energy between the two adsorbed layers, at least in those cases in which the gas molecules do not possess considerable permanent dipole moments [R.C. Bansal, et al. 2005].

The basic assumption of the BET theory is that the Langmuir Isotherm can be applied to every adsorption layer. The theory postulates that the first layer of adsorbed molecules acts as a base for the adsorption of the second layer of molecules and so on, so that the concept of localization is maintained in all layers. Furthermore, the forces of interaction between the adsorbed molecules are also neglected, as in the case of Langmuir concept [R.C. Bansal, et al. 2005].

The resulting BET equation is expressed by (1):

$$\frac{1}{v\left[(P_0/P)-1\right]}=\frac{c-1}{v_m c}\left(\frac{P}{P_0}\right)+\frac{1}{v_m c} \qquad (2.1)$$

where P and P_0 are the equilibrium and the saturation pressure of adsorbates at the temperature of adsorption, v is the adsorbed gas quantity, and v_m is the monolayer adsorbed gas quantity. c is the BET constant, which is expressed by (2):

$$c = \exp\left(\frac{E_1 - E_L}{RT}\right) \tag{2.2}$$

where E_1 is the heat of adsorption for the first layer, and E_L is that for the second and higher layers and is equal to the heat of liquefaction.

Equation (1) is an adsorption isotherm and can be plotted as a straight line with $1 / v[(P_0 / P) - 1]$ versus P / P_0 according to experimental results. The value of the y-intercept is used to calculate the monolayer adsorbed gas quantity v_m and the BET constant c.

A total surface area S_{total} and a specific surface area S are evaluated by the following equations:

$$S_{BET,total} = \frac{(v_m N s)}{V} \tag{2.3}$$

$$S_{BET} = \frac{S_{total}}{a} \tag{2.4}$$

where N is the Avogadro's number, s is the adsorption cross section, V is the molar volume of adsorbent gas, and a is the molar weight of adsorbed species [wikipedia.org].

Refer to M. Z. Figueroa-Torres, et al., adsorption isotherm can be plotted from the BET data and all isotherms produced from the activated carbon shows Type I. According to R.C. Bansal, et al., when a solid contains very fine mircropores that have pore dimension only a few molecular diameters, the potential field of force from the neighboring walls of the pores will overlap causing an increase in the interaction energy between the solid surface and the gas molecules. This will result in an increase in adsorption, especially at low relative pressures. There is a possibility that the interaction energy may be large enough to bring about complete filling of the pores at quire low relative vapor pressure, giving rise to a Type I isotherm. Type I isotherms are generally common to chemisorption, although some physical adsorption such as those in highly microporous active carbons and in carbon molecular sieves also conform to this Type.

Nitrogen adsorption isotherms were used for calculating surface area and pore volume through BET equation and Gurvitsch rule [H. Jin, et al. 2007]. It is also well known that Horvath-Kawazoe (HK) equation is quite excellent for measuring the volume of micropore [J.S. Im, et al. 2008]. Dubinin-Radushkevich (DR) equation can also be used to calculate the micropore volumes from the adsorption isotherm [M. Z. Figueroa-Torres, et al. 2007].

2.4.3 Hydrogen Adsorption Capacity Analysis

Study on the amount of hydrogen gas that can be stored into the developed adsorbent is then to be conducted. According to the research done by J. S. Im, et al., there are three general different techniques applied in hydrogen adsorption capacity analysis:

- Volumetric method
- Gravimetric method
- Thermal desorption spectroscopy

Volumetric method and gravimetric method are commonly used as state in the journals. Both methods require the samples to be degassed at certain temperature. It is suggested that the samples are to be degassed at 473K for 2 hours and conduct hydrogen adsorption at 303K and 0 – 30 atm for hydrogen adsorption capacity analysis using volumetric method [J. S. Im, et al. 2008].

In volumetric method, pressure, temperature and composition is measured as a function of time. There is pressure change arising from system temperature variation can incorrectly be interpreted as adsorption in volumetric adsorption apparatus which need to be avoided. Alternatively, gravimetric method will be used, where the pressure, temperature and weight are monitored as a function of time. The measured value is caused by the hydrogen uptake on activated carbon and buoyancy force. As the buoyancy increases according to the increase of adsorption pressure and its effect was larger than the hydrogen uptake, the measured value decreased in the opposite direction of pressure [R. L. Tseng, 2006]. In this study, Magnetic Suspension Balance using gravimetric method can be used to perform the hydrogen adsorption capacity analysis.

CHAPTER 3
METHODOLOGY

Figure 3.1 shows the process flow of the research work. In this research work, sugarcane bagasse chosen as a precursor will be prepared and undergoes the chemical activation using KOH, NaOH, and $ZnCl_2$ solution. Different chemical activation is to compare the effect of different activation agent on development of porosity and surface roughness. Further more, the samples will undergo the carbonaceous step by using "Fixed Bed Activation Unit" (FBAU) before being characterized using SEM, and Energy Dispersive X-Ray (EDX) analysis. After all, the result will be analyzed and to be compared with the commercial carbon activated for hydrogen storage.

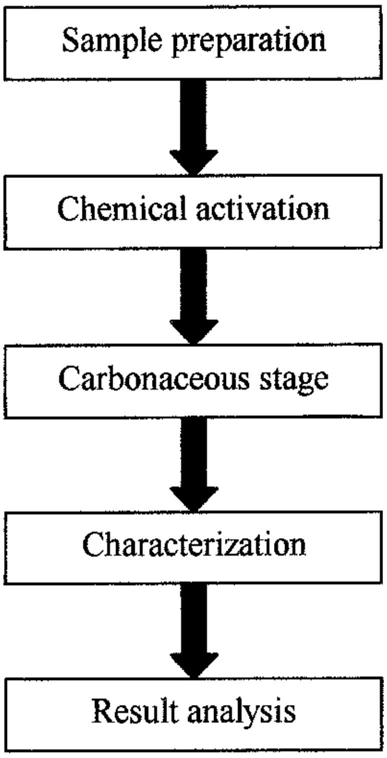

Figure 3.1: Process flow chart of the research work

3.1 Chemical Activation

Activating agent is to remove impurities to form carbon with extremely porous structure, which is activated carbon [Activating carbon particles, www.aquatechnologies.com]. The activating agents that most commonly used are phosphoric acid, zinc chloride, and alkaline metal compounds such as potassium hydroxide and sodium chloride. Phosphoric acid and zinc chloride are used for the activation of lignocellulosic materials, which have not been carbonized previously; whereas metal compounds such as KOH are used for activation of coal precursors or chars [C. Srinivasakannan, et al. 2004]. Literature done by R.L. Tseng revealed that physical method is better than impregnation method. However, during the commercial production of activated carbon, physical mixing needs particulate of chemical, while the impregnation method can use concentrated chemical liquor. Because of more convenient storage, transportation, and lower cost, chemical liquor is preferred for industrial production.

In chemical activation step, KOH, NaOH, and $ZnCl_2$ will be used with different concentration of 2M, 4M and 6M. The effect of the different activating agent to the pore distribution will then analyze. Higher concentration of activating agent will increase the reaction rate and then attribute to excessive carbon burn-off, resulting in widening of pore diameter and the collapse of some pore walls between pores, and thus decrease the specific surface area and micropore area [H. Jin, et al. 2007].

The detailed procedure for chemical activation will be discussed in the following section:

3.1.1 Preparation of KOH, NaOH, and $ZnCl_2$ Solution

i. 2M, 4M and 6M of KOH, NaOH, and $ZnCl_2$ solution as activating agent are prepared. The following equation is used to obtain the weight of chemical pellets in order to produce the 1L solution with concentration respectively as shown in Table 3.2.

$$W = \text{Molarity x Molecular Weight}$$

Table 3.1: Molecular weight of KOH, NaOH, and $ZnCl_2$

Activating Agent	Molecular Weight (g/mol)
KOH	56.11
NaOH	39.99
$ZnCl_2$	136.28

Table 3.2: Amount required for preparation of 1L solution for various activating agent

Concentration (M)	Weight of KOH (g)	Weight of NaOH (g)	Weight of $ZnCl_2$ (g)
2	112.22	79.98	272.56
4	224.44	159.96	545.12
6	336.66	239.94	817.68

3.1.2 Chemical Impregnation

i. 3 samples of 20 g sugarcane bagasse precursor are weighted.

ii. Each sample is impregnated in 200 ml of 2M, 4M, and 6M of KOH solution. Samples are impregnated overnight so that the reagent is fully adsorbed into the raw material.

Figure 3.2: Impregnation of precursors with KOH

iii. The samples are then filtered and dried in the oven overnight at 110°C to evaporate the remaining water.

iv. Samples are ready for carbonaceous step.

v. Step (i) to (iv) is repeated for the impregnation of sugarcane bagasse with NaOH and $ZnCl_2$ activating agent.

3.2 Carbonaceous Stage

Carbonaceous stage which include dry and heating (400°C to 600°C) in oxygen-deficient atmosphere that cannot support combustion will take place after the chemical activation, to separate by product also to drive off any gases generated [Activating carbon particles, www.aquatechnologies.com]. The chemically activated samples will put into Fixed Bed Activation Unit (FBAU) with the horizontal combustion chamber orientation at pyrolysis temperature. In this carbonaceous step, variables of pyrolysis temperature, pyrolysis duration, type of gas flow and gas flow rate are studied. Nitrogen gas flow which is now available in the laboratory will be flowed during the soaking period. Yield of the activated carbon is then determined for the effect observation of activating agent with different concentration on the carbon material.

3.2.1 *Procedure of Carbonaceous Stage*

i. The chemically activated precursor is weighted as W_1 and put into crucible.

ii. Crucible with activated precursor is put into the horizontal chamber of FBAU.

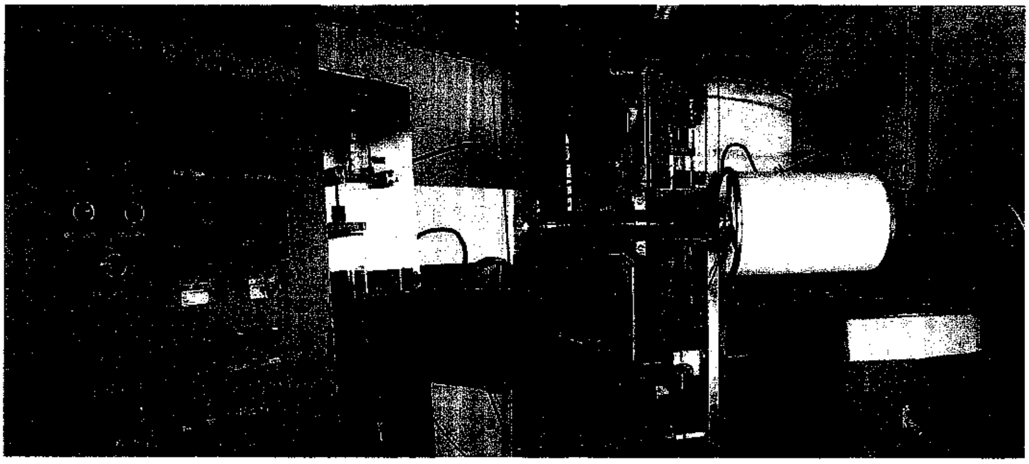

Figure 3.3: Fixed Bed Activation Unit with combustion chamber

iii. Nitrogen gas is flowed at 0.2 L/min.

iv. Pyrolysis temperature is set at 600°C and maintained for 3 hours soaking time.

v. After the soaking time, the sample is left inside the chamber until it is cooled to room temperature. Nitrogen gas is continued flowing at the mean time to prevent oxygen gasses from the atmosphere into the chamber.

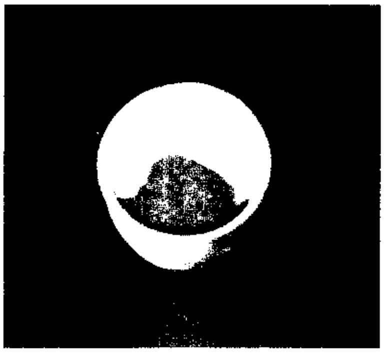

Figure 3.4: Activated Carbon after carbonaceous step

vi. The activated carbon is weighted as W_2 and the yield of the activated carbon is calculated.

$$\text{Yield} = W_2 / W_1$$

vii. The activated carbon is washed with distilled water to remove the remaining excessive chemical, and dried in the furnace overnight at 110°C.

viii. The adsorbent is now ready for characterization study.

3.3 Characterization

3.3.1 SEM and Energy Dispersive X-Ray (EDX) Analysis

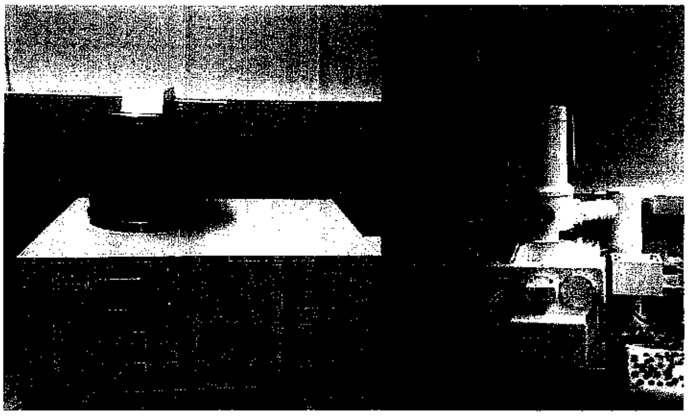

Figure 3.5: Scanning electron microscope with sputter coater

The activated carbon is undergone sputter coat with the gold palladium prior to SEM images. Samples are randomly selected to perform the EDX test to obtain the details of chemical components that exist on the produced adsorbent.

CHAPTER 4

RESULT AND DISCUSSION

The condition of chemical activation and carbonaceous step for each sample is summarized in Table 4.1.

Table 4.1: Summary of activation condition

Activating Agent	Molarity (M)	Pyrolysis Temperature (°C)	Pyrolysis Time (h)
No chemical Impregnation	-	600	3
$ZnCl_2$	2	600	3
$ZnCl_2$	4	600	3
$ZnCl_2$	6	600	3
KOH	2	600	3
KOH	4	600	3
KOH	6	600	3
NaOH	2	600	3
NaOH	4	600	3
NaOH	6	600	3

4.1 SEM Images

SEM images of the sugarcane bagasse activated by using $ZnCl_2$, KOH, and NaOH with molarity of 2M, 4M, and 6M, are shown in this section.

4.1.1 Precursor Sugarcane Bagasse

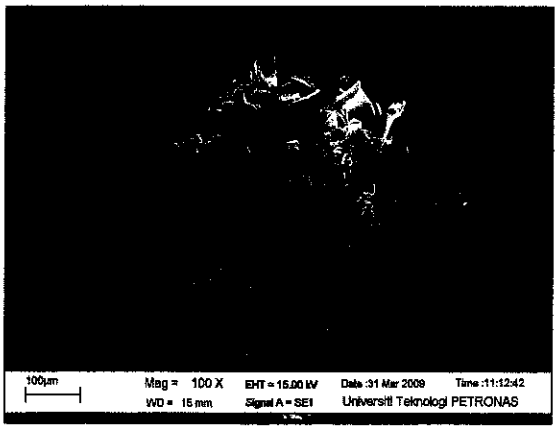

Figure 4.1: Sugarcane bagasse without chemical impregnation and carbonization

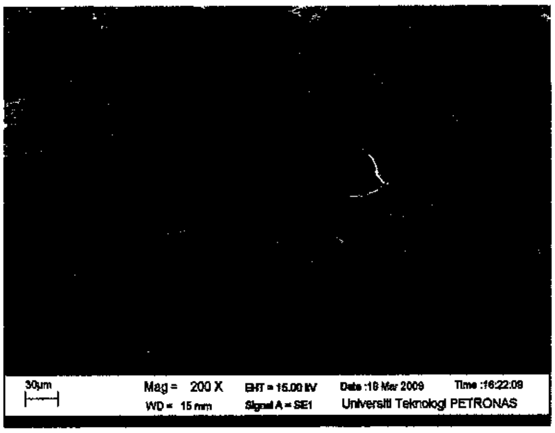

Figure 4.2: Carbonized sugarcane bagasse without chemical impregnation

4.1.2 $ZnCl_2$ Activated Carbon

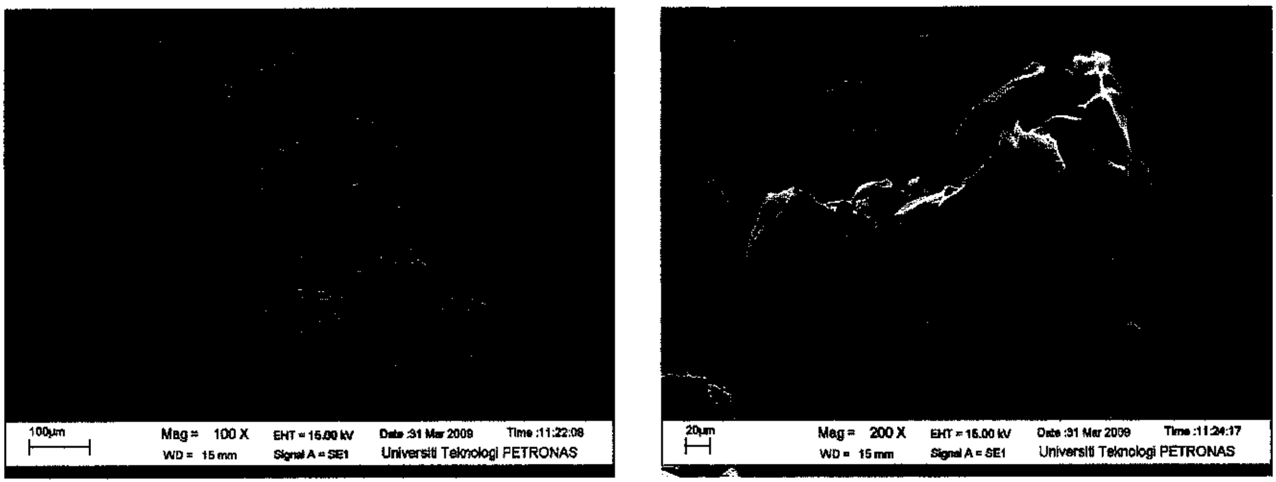

Figure 4.3: Carbonized sugarcane bagasse with 2M $ZnCl_2$ activation

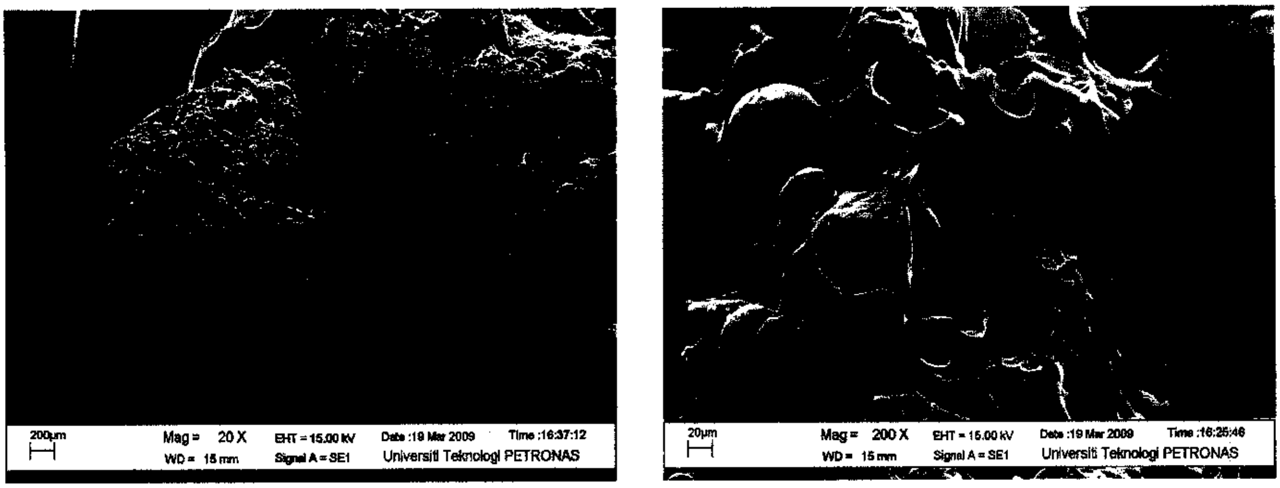

Figure 4.4: Carbonized sugarcane bagasse with 4M $ZnCl_2$ activation

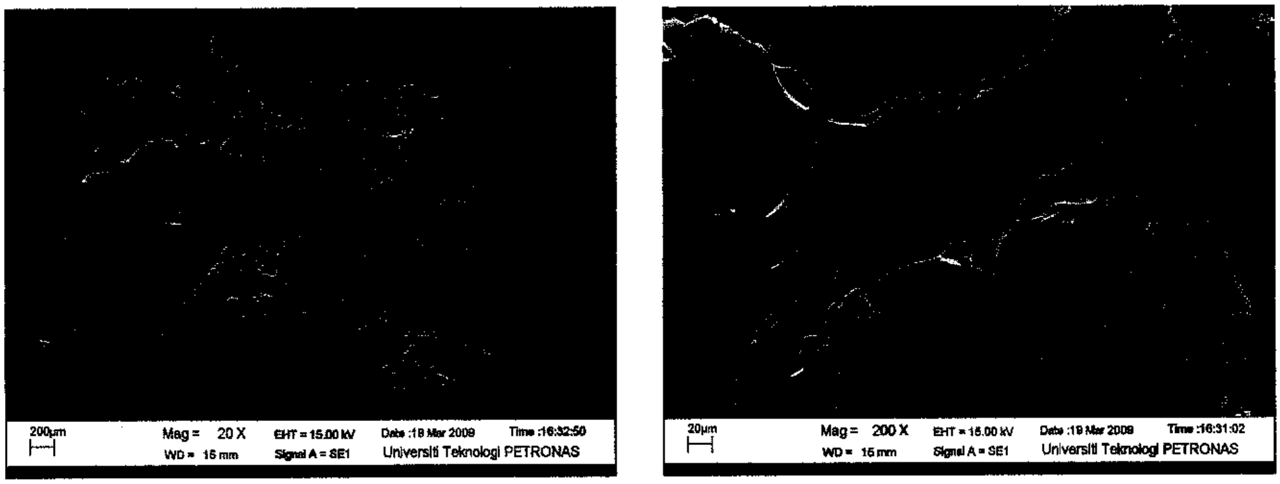

Figure 4.5: Carbonized sugarcane bagasse with 6M $ZnCl_2$ activation

4.1.3 KOH Activated Carbon

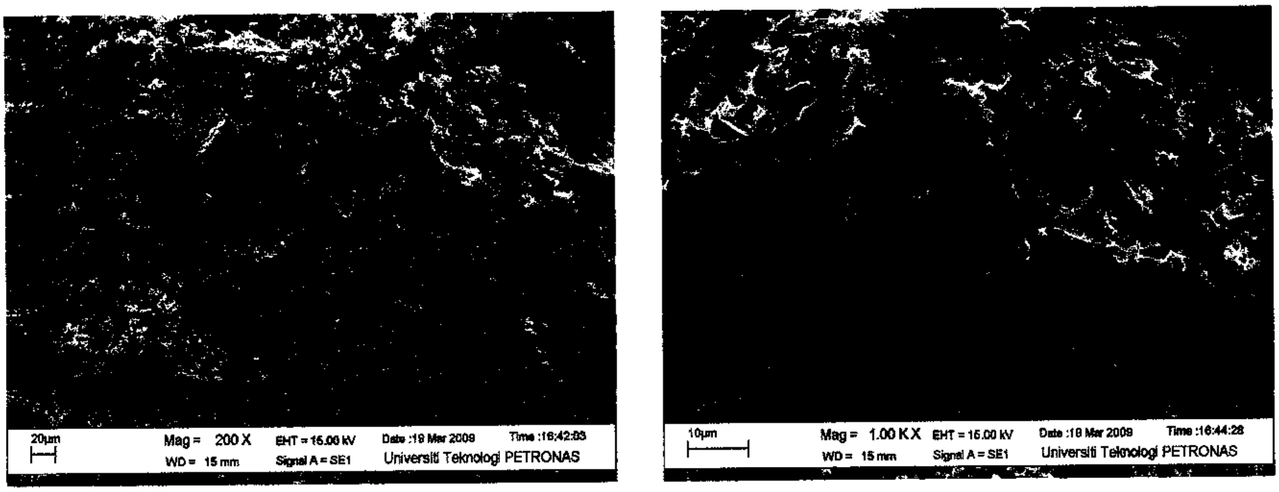

Figure 4.6: Carbonized sugarcane bagasse with 2M KOH activation

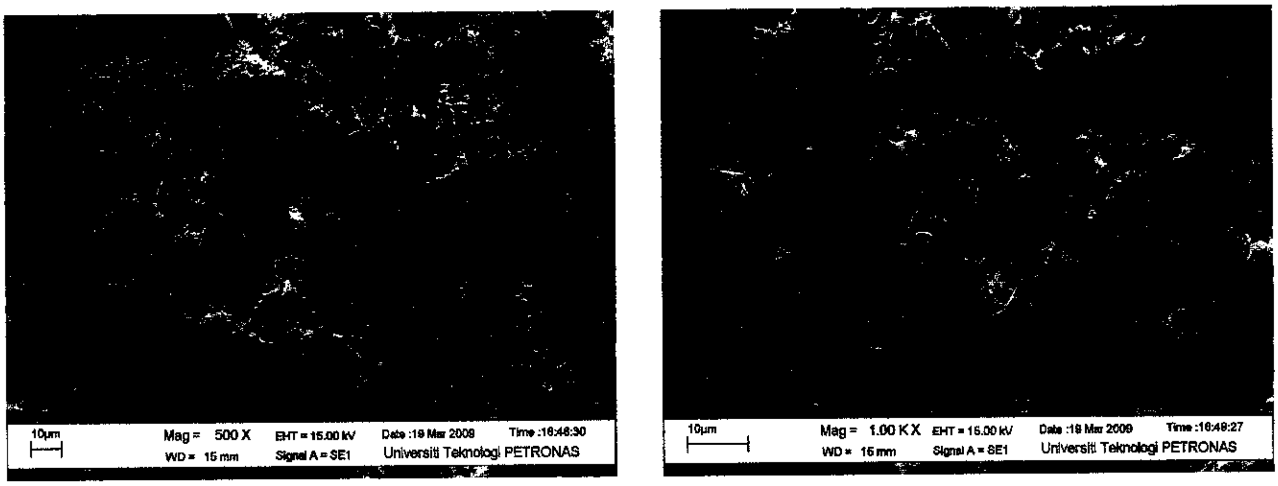

Figure 4.7: Carbonized sugarcane bagasse with 4M KOH activation

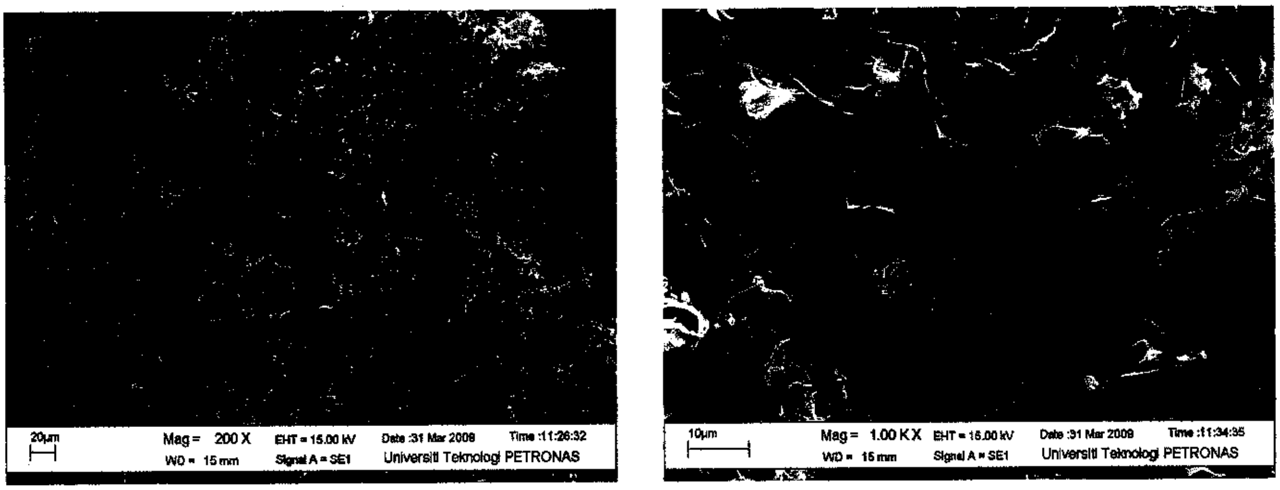

Figure 4.8: Carbonized sugarcane bagasse with 6M KOH activation

4.1.4 NaOH Activated Carbon

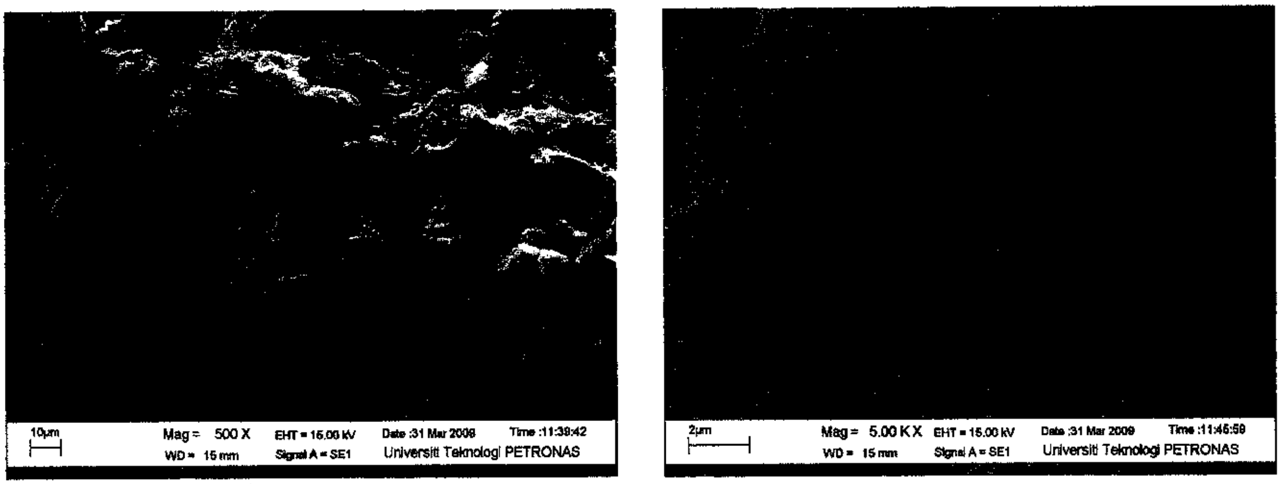

Figure 4.9: Carbonized sugarcane bagasse with 2M NaOH activation

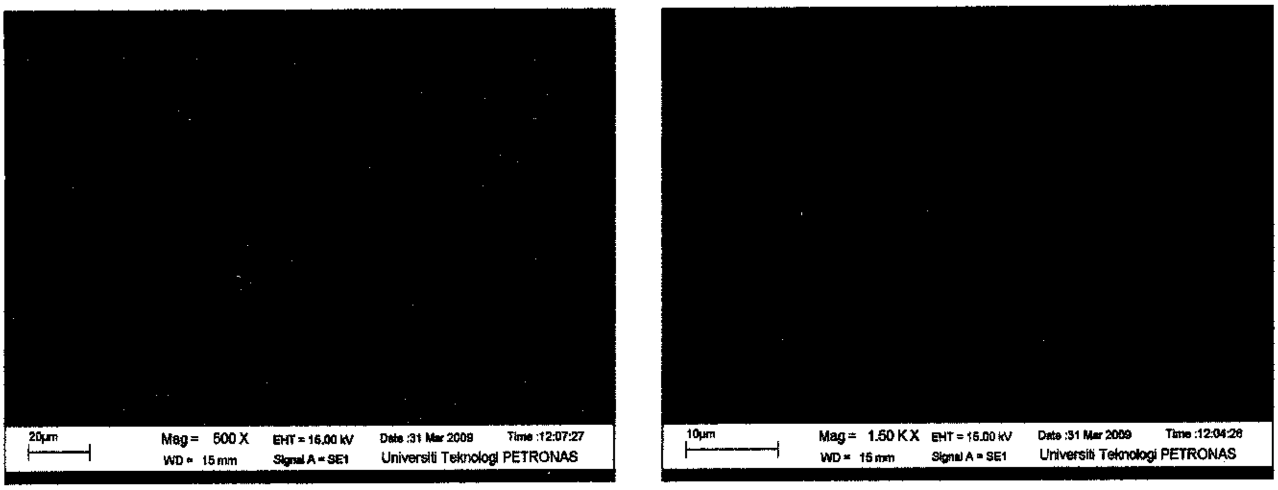

Figure 4.10: Carbonized sugarcane bagasse with 4M NaOH activation

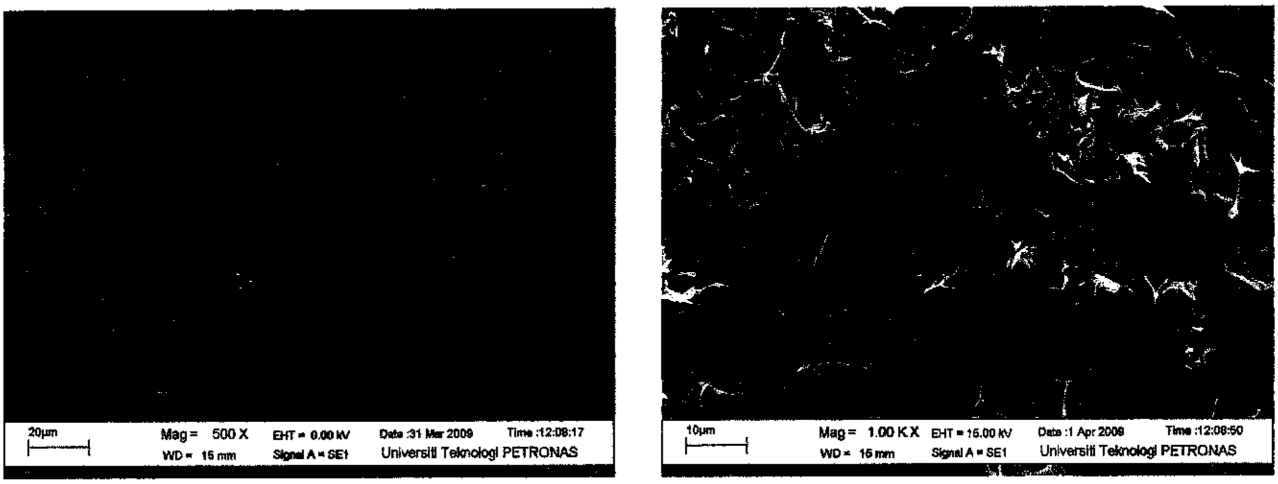

Figure 4.11: Carbonized sugarcane bagasse with 6M NaOH activation

4.2 Energy Dispersive X-Ray (EDX) Analysis

The results of chemical property measured included the elemental analysis for each activated carbon obtained from EDX analysis is displayed in Table 4.2.

Table 4.2: Element analysis for activated sugarcane bagasse

Activated Carbon	Element Analysis (%)
2M $ZnCl_2$	• C : 91.51 • Cl : 3.30 • Zn : 5.19
4M $ZnCl_2$	• C : 80.11 • O : 13.96 • Cl : 1.83 • Zn : 4.10
6M $ZnCl_2$	• C : 74.13 • O : 12.39 • Cl : 5.64 • Zn : 7.84
2M KOH	• C : 86.53 • O : 12.51 • K : 0.95
4M KOH	• C : 82.89 • O : 16.47 • K : 0.63
6M KOH	• C : 70.13 • O : 29.49 • K : 0.38
2M NaOH	• C : 86.80 • O : 11.91 • Na : 1.29
4M NaOH	• C : 68.24 • O : 26.52 • Na : 5.24
6M NaOH	• C : 56.16 • O : 33.75 • Na : 10.09

4.3 Observation Obtained during Experiment

During the of $ZnCl_2$ activation, the impregnated precursor materials are found to be black in color after dried over night at 110°C. Same result obtained by C. Srinivasakannan where the impregnated precursor material is found to blacken and form a plastic mass during the first stage of activation carried out at 200°C for 15 min, which it has ultimately transformed to a dry char. A white soluble product was found when washing the NaOH activated samples with distilled water. Refer to R.L. Tseng, the white soluble product sparked with crack when in contact with humid air or water, which proved the existence of Na after the reaction.

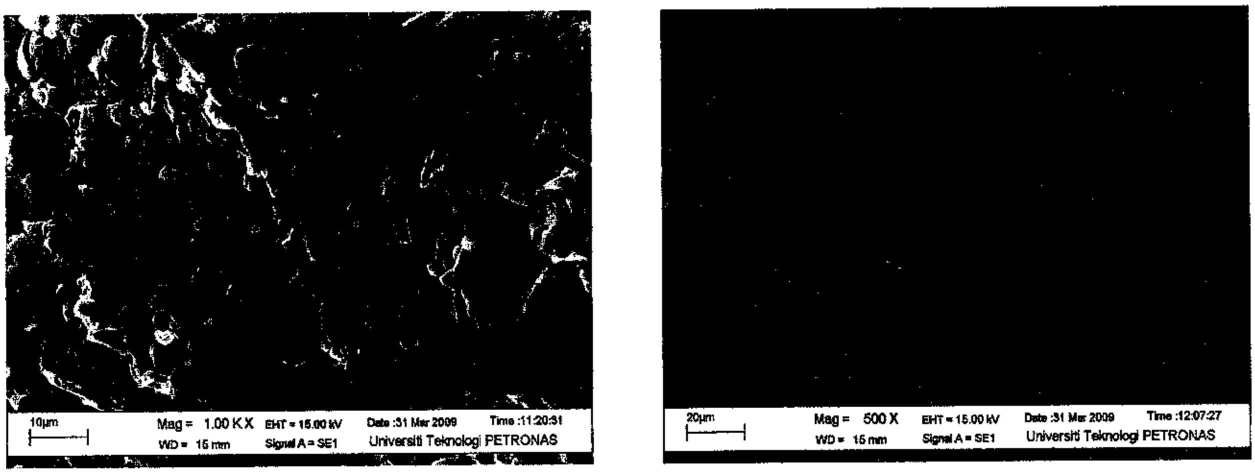

Figure 4.12: SEM images of 4M NaOH activated carbon (a) without washed by distilled water, and (b) washed with distilled water

Both washing and without washing after the carbonization step as shown in Figure 4.12 is compared. It is obvious that the activated carbon without washed by distilled water shows the pores of the carbon is covered by a thick coating. The coating is observed to cover the edges of the pores and clogged the porosity. This may due to the left over of chemicals on the carbon surface during chemical impregnation. It is supported by the element analysis of both washing and without washing as shown in Table 4.3, where the weight percent of Na in the activated carbon without washing is 27.19 wt%, and then reduced to 5.24 wt% after washing with distilled water. Refer to C. Srinivasakannan and H. Jin, the step of washing the product with distilled water is necessary to remove the remaining excessive chemicals.

Table 4.3: Comparison of element analysis of activated carbon with and without washed

	4M NaOH activated carbon (without washed)	4M NaOH activated carbon (washed)
Element analysis	• C : 15.30 wt% • O : 57.51 wt% • Na : 27.19 wt%	• C : 68.24 wt% • O : 26.52 wt% • Na : 5.24 wt%

4.4 Effect of Chemical Activation on the Carbon Content

Weight percentage of elements exist in the samples is obtained from the EDX analysis as shown in Table 4.2. Beside the content of carbon, the activated carbon is also consists of the elements that left over from activating agents respectively during impregnation. The graph of carbon content versus concentration of various activating agent is plotted and shown in Figure 4.13.

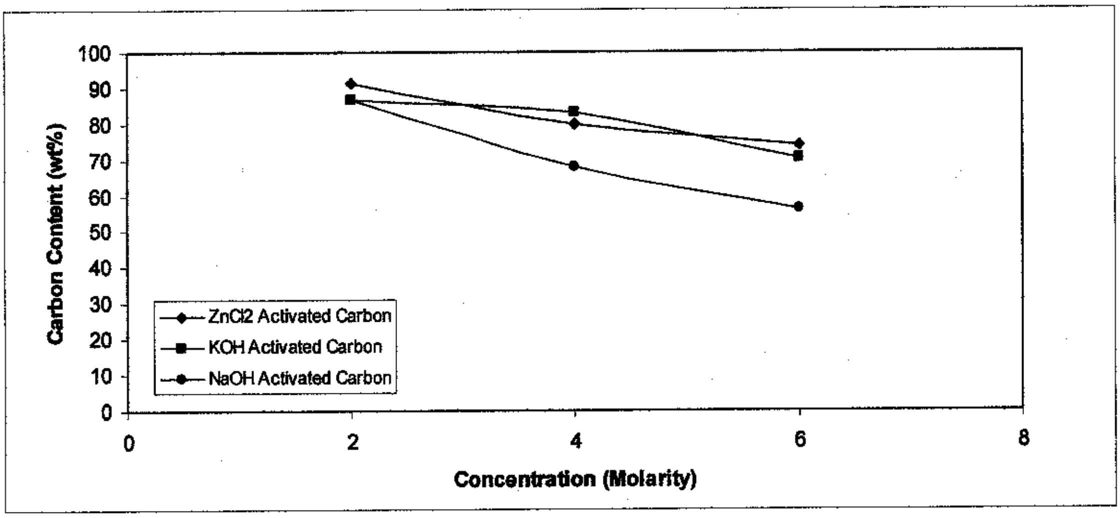

Figure 4.13: Carbon content for various activating agent in the relation of concentration

From the graph, the carbon content is reduced as the concentration of activating agents increased. This explains the effect of chemical activation on the carbon surface, which removes the impurities to form carbon with extremely porous structure. It is observed that the KOH and NaOH activation give lower carbon content compared to $ZnCl_2$ activation. This may due to the effect of oxygen in the presence of KOH and NaOH solutions that will lead to carbon burn off from outside. This explanation is supported by J.S. Im.

Among the three activating agents, NaOH activation gives the lowest carbon content. This result obtained is consistent with the study of mesopore control of high surface area NaOH activated carbon done by R.L Tseng, where it is stated that the product of Na from NaOH reaction with C kept undergoing oxidation and hydration with O and H in the char, forming NaOH again and reacting with C in the char.

4.5 Effect of Chemical Activation on the Surface Morphology

The SEM images show that the porosity developed for $ZnCl_2$, KOH, and NaOH activation of carbons are all exhibited in spongy structure. Figure 4.3 to Figure 4.5 shows the SEM images for $ZnCl_2$ activated carbon and non uniform of surface roughness and porosity development is obtained. The pore development is higher at 6M of $ZnCl_2$ activation compared to the pore development at 2M of $ZnCl_2$ activation. Refer to J.S. Im, this may due to higher concentration of $ZnCl_2$ will dissolve more in carbon and thus enlarging the pore diameter.

It is obvious that the porosity development for $ZnCl_2$ activation is less efficient compared with KOH and NaOH activation. J.S. Im suggested the different activation behavior to generate the porosity, since the $ZnCl_2$ dissolved it in bagasse fiber forming uniform porosity, whereas for the condition under KOH activation carbon were burn off from outside due to oxygen of KOH onto the carbon surface. On the other hand, M. Z. Figueroa-Torres suggested that the activation of these materials takes place as a result of chemical reaction between carbon and potassium or sodium hydroxide under nitrogen atmosphere, where the main reaction consists of hydroxides reduction and carbon oxidation. Thus, the porosity is formed by the release of carbon monoxide, carbon dioxide, and hydrogen gas.

Figure 4.6 to Figure 4.8 shows the SEM images for KOH activated carbon. The surface roughness of KOH activated carbon is not that smooth and the diameter of porosity developed is non-uniform in same activation and pyrolysis condition. It is observed that the diameter of pore developed is increased with the increasing of KOH solution molarity from 2M to 6M, forming "bone-like" structure as shown in Figure 4.8. J.S. Im suggested that it is due to the effect of oxygen in the presence of KOH solutions which lead to excessive carbon burn off, resulting in the widening of pore diameter and the collapse of some pore walls between pores that will reduce the specific surface area and micropore surface area. This explanation is also supported by H. Jin.

Figure 4.9 to Figure 4.11 shows the SEM images for NaOH activated carbon, which give a smoother porosity development compared with KOH activation. Same observation of "bone-like" structure formation is obtained at the activation of 6M NaOH as shown in Figure 4.11. The pore diameter is increased as the concentration of NaOH activation is increased, which is due to the effect of oxygen in the presence of NaOH onto the carbon surface. Beside, R.L. Tseng is suggested that the product Na of reaction NaOH with C kept undergoing oxidation and hydration with O and H in the char and forming NaOH again and reacting with C in the char, which could enlarge the pore diameter and the collapse of pore walls between pores, thus forming "bone-like" structure.

J.S. Im suggested that KOH activation is much more effective to increase specific surface area and total pore volume than $ZnCl_2$ activation. However, in case of micropore volume which has pore diameter from 0.6 to 0.7 nm, $ZnCl_2$ is much more effective than KOH activation due to the different activation behavior between $ZnCl_2$ and KOH to generate the porosity. While H. Jin suggested that KOH has given an excellent performance as chemical activating agent, which could lead to a very high specific surface area and micropore surface area, and linearly increases the hydrogen adsorption capacity. On the other hand, R.L. Tseng suggested that the reaction mechanism of NaOH is similar to that of KOH and that the same high surface area activated carbon could be obtained using NaOH activation as KOH activation. This explained the similar "bone-like" structure is obtained for both 6M of KOH and NaOH activations in the result. From the SEM images, NaOH activation gives the better surface morphology to sugarcane bagasse. Research done by R.L. Tseng revealed that using NaOH activation in activated carbon preparation is useful. The reasons are summarized as follows:

- The molecular weight of KOH (56.1 g/mol) is larger than that of NaOH (40.0 g/mol). Thus the dosage (weight measurement) of NaOH activated carbon is less than that of KOH activated carbon.
- NaOH is cheaper than KOH.
- Based on life cycle assessment, NaOH is more environmentally friendly.
- NaOH is less corrosive than KOH. The equipment for NaOH activation would last longer.

4.6 Influence of Pore Width on Hydrogen Adsorption

As reported in literature review, pore width is one of the important factors that affect the adsorption of hydrogen. As shown in Figure 4.14, the dynamic hydrogen molecular would not enter to the inside of pore due to insufficient pore width. In case of too much bigger pore width comparing with the diameter of dynamic hydrogen molecule, the dynamic hydrogen molecule could go outside easily even though it could be in pore [J.S. Im, et al. 2008]. So it is difficult to trap hydrogen molecule inside the pore which result in insufficient hydrogen adsorption. The diameter of dynamic hydrogen molecule is 0.4059 nm. Thus, J. S. Im suggested that the most effective factor which can attract the hydrogen adsorption capacity positively is the pore volume in the range of pore width mainly from 0.6 to 0.7 nm.

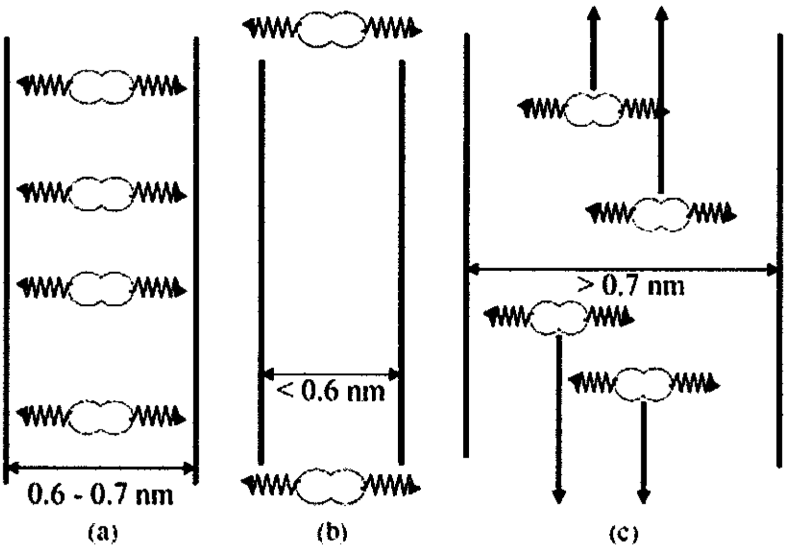

Figure 4.14: Mechanism of hydrogen adsorption: (a) 0.6 nm < pore width < 0.7 nm; (b) pore width < 0.6 nm; (c) pore width > 0.7 nm

CHAPTER 5
CONCLUSION

Hydrogen is a perfect fuel for future energy application with number of advantages and most importantly, it contributes in reduction of greenhouse gas emissions. Thus, an efficient hydrogen storage medium is crucial for the adsorption of hydrogen as an energy carrier. A study of adsorbent synthesized from agriculture waste is carried out in order to develop an improved activated carbon as adsorbent for hydrogen storage. These unwanted agriculture waste which were generated in huge amount annually, can be converted into value-added material such as activated carbon.

Some literature review and study on researches done in the field activated carbon for hydrogen storage is carried out and reported. There are several factors that affect the adsorption capability of an adsorbent are nature or raw material, degree of activation and carbonaceous process, and the properties of adsorbent. Evaluation of properties on adsorbent is based on porosity developed, pore volume, pore size distribution, surface roughness, and density. The adsorption capability is highly dependent on the mentioned properties, which rely on the degree of activation and carbonaceous process, also the nature of the raw material. The variable keys in activation process are the type of activating agent, concentration of activating agent, duration of activation period, and the way of mixing or impregnation. While the variable key in carbonaceous process is the pyrolysis temperature, pyrolysis duration, type of inert flow and its flow rate.

Activated carbon developed from agriculture waste of sugarcane bagasse activated using $ZnCl_2$, KOH and NaOH is produced. Parameters being studied include the type of activating agent and its concentration. In order to study the effect of concentration on porosity development, the concentration of activating agents is varied from 2M to 6M, with constant variables in carbonaceous process of pyrolysis temperature at 600°C for 3 hours in 0.2 L/min nitrogen flow.

The step of washing the product with distilled water is necessary to remove the remaining excessive chemicals. From SEM images, the carbon activated by KOH and NaOH show the uniform and improved surface morphology and resembles the spongy structure. The $ZnCl_2$ activation also gives the spongy structure of porosity, but the pores structure obtained is not uniform. The increasing of activating concentration leads to excessive carbon burn off that widening the pore diameter and collapse of pore walls between pores, which could reduce the specific surface area and linearly reduce the hydrogen adsorption capacity, as stated by J.S. Im and H. Jin. The result is also supported by the element analysis result which shows that the carbon content is decreased with increasing of molarity of activating agents. NaOH activation gives the lowest carbon content among the activating agents, due to the product sodium reacted with O and H back to NaOH and react with C again as stated by R.L. Tseng.

Activated carbons are high in porosity, high surface area materials manufactured by carbonization and activation of carbonaceous materials, hence high surface capacity for adsorption [C. Srinivasakannan, et al. 2004]. This is also supported by H. Jin, et al., as the hydrogen capacity increases almost linearly according to their specific area, also linearly related to their porosities such as surface area and pore volume. The results show that the activated carbons are high in development of porosity, thus higher hydrogen adsorption capacity could be obtained. However, the continuation for the current research work can be done by sending the samples for further analysis of specific surface area using BET apparatus and Magnetic Suspension Balance for hydrogen adsorption capacity.

CHAPTER 6

RECOMMENDATION

Every stage of the activated carbon development from sample preparation, chemical impregnation, to carbonaceous stage is crucial in guaranteeing that the success of synthesize of the adsorbent. Some recommendations are provided in order to improve the current research work.

6.1 Chemical Activation

The precursor should be immediately sent for carbonaceous stage right after the completion of chemical impregnation. This is because of the possibility of chemical reaction on the carbon surface and attribute to the additional carbon burn off resulting in the widening of pore diameter and collapse of some pore walls between pores due to over duration of impregnation process. Even though the impregnated precursor is dried, the moisture in the air could cause the samples to be impregnated in more then the overnight duration, which is beyond the methodology proposed.

Smaller sample size of concentration for activating agent should be carried out in identifying the optimum concentration for the porosity development. Beside, the research work with other chemical activating agents should be performed to discover the best activating agent for development of the high quality of adsorbent for hydrogen storage.

6.2 Carbonaceous Stage

FBAU is used for the implementation of carbonaceous step. It is important to ensure the precursor is placed in the right region of the combustion chamber. Incomplete combustion or lower pyrolysis temperature experienced by the precursor might occur if the precursor is placed out of the mentioned region. The step of allowing the nitrogen flow for 5 minutes is needed to ensure the fully inert atmosphere in the combustion chamber before the temperature of the furnace is raised.

It is suggested that manipulating on the pyrolysis temperature, pyrolysis duration, and the flow rate of inert flow for carbonaceous stage should be performed to obtain the best quality of adsorbent as these variables could affect the porosity development in the adsorbent.

After the carbonaceous step, it is necessary to wash the product with distilled water to remove the remaining excessive chemicals and then dried overnight. Extra attention should be paid during the washing step to avoid the possibility of crunching the activated carbon. This could destroy the pore structure and collapse of pore walls between pores, result in non-uniform manner.

6.3 Characterization of Activated Carbon

Due to unavailability and limitation of the BET apparatus and Magnetic Suspension Balance throughout the research duration, characterization on specific surface area and hydrogen adsorption capacity is aborted. In order to develop a more convincing or promising adsorbent, further characterization on specific surface area and hydrogen adsorption capacity analysis should be performed to investigate the characteristic of the activated carbon produced.

6.4 Comparison with Commercial Adsorbent

In order to develop a more promising adsorbent, it is suggested to compare the developed adsorbent with the commercial adsorbent available for hydrogen storage. Comparison in terms of properties of adsorbent such as porosity development, surface roughness and hydrogen adsorption capacity, economic analysis, environmental impact, and safety issues should be carried out.

6.5 Marketability Concern

It is important to understand the effect of heating rate on the quality of the product. This leads to few complications while attempting to scale up the process as the optimum condition for good quality product is always reported in combination of activation time and temperature. If the temperature progression as reported in the lab scale experiments is important to obtain same quality of product, scare up of the process becomes very complicated with requirement of sophisticated controls to follow the temperature progression as in lab scale experiment adding up to the cost of production. It is advantageous for process economics to follow the maximum heating rate since processing time and size of the equipment could be reduced, which leads to economizing the process.

REFERENCES

C.Srinivasakannan, Mohamad Zailani Abu Bakar, "Production of activated carbon from rubber wood saw dust", Biomass and Bioenergy, 27 (2004) 89-96

H.Jin, Y.S.Lee, I.Hong, "Hydrogen adsorption characteristic of activated carbon", *Catalysis Today*, 120 (2007) 399-406

J. S. Im, S.J. Park, T.J Kim and Y. S. Lee, "The study of controlling pore size on electrospun nanofibers for hydrogen adsorption", *Journal of Colloid and Interface Science*, 318 (2008) 42-49

Joseph M. Norbeck, et al., 1996, *Hydrogen fuel for surface transportation*, Warrendale, PA : Society of Automotive Engineers, Inc.

M.Sharon, T.Soga, R.Afre, D.Sathiyamoorthy, K.Dasgupta, S.Bhardwaj, S.Jaybhaye, "Hydrogen storage by carbon materials synthesized from oil seeds and fibrous plant materials", *International Journal of Hydrogen Energy,* 32 (2007) 4238-4249

M. Z. Figueroa-Torres, A. Robau-Sanchez, L. De la Torre-Saenz, A. Aguilar-Elguezabal, "Hydrogen adsorption by nanostructured carbons synthesized by chemical activation", Microporous and Mesoporous Materials, 98 (2007) 89-93

N. Texier-Mandoki, J. Dentzer, T. Piquero, S. Saadallah, P. David and C. Vix-Guterl, "Hydrogen storage in activated carbon materials: Role of the nanoporous texture", *Carbon*, 42 (2004), 2744-2747

Peter Hoffmann, Tom Harkin, 2002, *Tomorrow's Energy: Hydrogen, Fuel Cells, and the Prospects for a Cleaner Planet*, MIT Press, 2-6

R.C.Bansal, M.Goyal, 2005, *Activated Carbon Adsorption*, CRC Press, Taylor & Francis, 1-144

R.L.Tseng, "Mesopore control of high surface area NaOH activated carbon", *Journal of Colloid and Interfacial Science*, 303 (2006) 494-502

Tan Rui Yee, 2008, *Synthesis and Characterization of Adsorbent for Hydrogen Storage*, Undergraduate Thesis, Chemical Engineering, Universiti Teknologi Petronas, Malaysia

Activating carbon particles. Retrieved September 20, 2008, from <http://www.aquatechnologies.com/info_activated_carbon.htm>

Adsorbent. In *Wikipedia, The Free Encyclopedia.* Retrieved August 6, 2008, from <http://en.wikipedia.org/wiki/Adsorption>

Bagasse. In *Wikipedia, The Free Encyclopedia.* Retrieved October 13, 2008, from <http://en.wikipedia.org/wiki/Bagasse>

BET theory. In *Wikipedia, The Free Encyclopedia.* Retrieved August 6, 2008, from <http://en.wikipedia.org/wiki/BET_theory>

Heat of combustion for common fuels. Retrieved August 6, 2008, from <http://en.wikipedia.org/wiki/Heating_value>

Hydrogen Storage. Retrieved August 6, 2008, from <http://www.rsc.org/delivery/_ArticleLinking/DisplayHTMLArticleforfree.cfm?JournalCode=EE&Year=2008&ManuscriptID=b807957a&Iss=Advance_Article>

Proceedings of the Fiji/FAO 1997 Asia Pacific Sugar Conference. Retrieved October 13, 2008, from <http://www.fao.org/docrep/005/x0513e/x0513e22.htm>

Sugarcane. In *Wikipedia, The Free Encyclopedia*. Retrieved August 12, 2008, from <http://en.wikipedia.org/wiki/Sugar_cane>

The Hydrogen Economic. Retrieved August 6, 2008, from <http://www.americanenergyindependence.com/hydrogen.html>