POST-COMBUSTION CARBON DIOXIDE CAPTURE BY POROUS CARBON FROM CASSAVA LEAVES IN A FIXED-BED

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LIST OF SYMBOL

Symbol	Description	Unit
C_o	Inlet concentrations of CO_2	%
C	Outlet concentrations of CO_2	%
F_o	Volumetric flowrate of CO_2	ml/min
m	Mass of adsorbent in the adsorption column	g
M_w	Molecular weight of CO_2	mg/mmol
q_e	Adsorption capacity	mmol/g
t	Adsorption time	min
W_f	weight of adsorbent after the process	g
W_i	weight of precursor at initial stage	g
ρ_{CO_2}	Density of CO_2	mg/ml

LIST OF ABBREVIATION

CCS	Carbon dioxide capture and storage
CL	Cassava leaves
CO_2	Carbon Dioxide gas
EDX	Energy Dispersive X-Ray
FTIR	Fourier Transform Infrared Spectroscopy
H_2S	Hydrogen Sulphide
H_3PO_4	Phosphoric Acid
KOH	Potassium Hydroxide
N_2	Nitrogen gas
NaOH	Sodium Hydroxide
rpm	Rotation per minute
SEM	Scanning Electron Microscope
$ZnCL_2$	Zinc Chloride

PENJERAPAN KARBON DIOKSIDA SELEPAS PEMBAKARAN MENGGUNAKAN KARBON BERLIANG DARI DAUN UBI DALAM LAPISAN TETAP

ABSTRAK

Karbon berliang telah mendapat perhatian dalam penyerapan karbon dioksida. Ciri fizikal dan kimianya boleh ditingkatkan melalui beberapa pengubahsuaian dengan mengaktifkannya dengan agen pengaktif. Dalam kajian ini, daun ubi kayu digunakan sebagai bahan mentah untuk menyediakan karbon berliang bagi menjerap karbon dioksida. Daun ubi kayu dipilih kerana ia adalah bahan sisa, mudah didapati dan murah. Karbon berliang telah disediakan melalui beberapa langkah pengaktifan karbon dan pengaktifan kimia. Morfologi permukaan dan kumpulan berfungsi penjerap yang disediakan telah dicirikan oleh luas permukaan BET, SEM dan FTIR. Penjerapan karbon dioksida dilakukan dalam sistem penjerapan dengan penganalisis karbon dioksida secara terus untuk menentukan kepekatan karbon dioksida di dalam gas. Kesan-kesan keadaan operasi seperti kadar aliran gas (30-60 mL/min), suhu penjerapan (30-60 °C) dan kepekatan karbon dioksida (10-20%) telah dikaji. Keluasan permukaan BET ialah 1246.07 m^2/g. Spektrum FTIR yang diperoleh untuk CL@600-K2 menunjukkan lebih banyak kumpulan fungsi amina berbanding CL@600. Kapasiti penjerapan terbaik dan masa tepu yang diperolehi adalah pada keadaan penjerapan kepekatan karbon dioksida sebanyak 10%, suhu penjerapan 30 °C, kadar aliran gas 30 mL/min dengan muatan penyerap 0.5 g masing-masing adalah 3.16 mmol / g dan 9.83 min. Keupayaan penjerapan tertinggi 4.54 mmol / g dicapai pada 20% kepekatan karbon dioksida. Penjanaan semula mampu melaksanakan sehingga 6 kitaran dengan penurunan 9.46% daripada kapasiti penjerapan awal. Keputusan yang diperoleh mencadangkan bahawa penjerap yang disediakan memberikan hasil positif terhadap prestasi penjerapan.

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ABSTRACT

Porous carbon has been gaining attention in carbon dioxide (CO_2) capture. Its physical and chemical characteristics can be enhance through some modifications by activating it with an activating agent. In this research, cassava leaves was used as a precursor to prepare adsorbents for CO_2 capture. Cassava leaves was chosen as it is waste, easily available and cheap. The adsorbents was prepared through a series of carbonization and chemical activation steps. The surface morphology and functional groups of the prepared adsorbents were characterized by Breneur Emmett Teller (BET) surface area, Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The adsorption of CO_2 was carried out in a fixed bed adsorption system with an online carbon dioxide analyzer to determine the concentration of CO_2 in the outlet gas. The effect of operating conditions such as total flow rate (30-60 mL/min), adsorption temperature (30-60 °C) and CO_2 concentration (10-20%) were studied. The BET surface area obtained is 1246.07 m^2/g. The FTIR spectra obtained for CL@600-K2 shows more amine functional group compared to CL@600. The best adsorption capacity and breakthrough time obtained is at adsorption condition of CO_2 concentration of 10%, adsorption temperature of 30 °C, total inlet flowrate of 30 mL/min with 0.5 g adsorbent loading was 3.16 mmol/g and 9.83 min respectively. The highest adsorption capacity of 4.54 mmol/g was achieved at 20% of CO_2 concentration. The regeneration was able to perform up to 6 cycles with a decreased of 9.46% from initial adsorption capacity. The results obtained suggest that the prepared adsorbent gave positive results on adsorption performance.

CHAPTER ONE

INTRODUCTION

1.1 Carbon Dioxide Capture

The consumption of water, food and energy is expanding as the world population grows and the rate of consumption cannot be sustained without depleting the planet's resources. As the demand of energy is increasing due to rapid economic growth, this also increase the consumption of fuel which as it is the key of energy sources particularly conventional fossil fuels (Leung et al., 2014). In order to satisfy the demands of energy consumption due to the population growth, the power plants need to increase the production of energy through more combustion of fossil-fuel.

Fossil fuels such as oil, coal, and natural gas combustions cater diverse purposes such as electricity generation, transportation, and industrial sector. However, burning of these fuels will release carbon dioxide (CO_2). The continual increased of CO_2 emission from fossil fuels may cause deteriorations of both environmental and ecosystem conditions (Rashidi and Yusup, 2016). Fossil fuels currently supply 80% of the world's energy need and the global energy demand is projected to be around 40% higher in 2035 than in 2010 (Global CCS Institute, 2014). The increase consumption of fossil fuels was believed as a major factor that increase the CO_2 release to the atmosphere.

CO_2 is a greenhouse gases that have the ability to absorb reflected solar radiation and trapped them in the atmosphere and this phenomenon is known as greenhouse effect (Raval and Ramanathan, 1989). The increase of CO_2 concentration in the ambient air is the primary contributor towards global warming scenario as it is the key element in the pace of temperature escalation. Over the last decade, global awareness about CO_2 emissions has increased, leading to increasing efforts to reduce their environmental impact, including preventive and remediation methods (Tan et al., 2014) and triggering the development of new technologies and materials for CO_2 capture and storage (CCS) (Chen et al., 2012). Table 1.1 shows the components of CCS.

Table 1.1: Components of CSS (Herzog, 2008)

Capture	The separation of CO_2 from an effluent stream and its compression to a liquid or supercritical state. Capture is generally required to be able to transport and store the CO_2 economically.
Transport	The movement of the CO_2 from its source to the storage reservoir. Transporting large quantities is most economically achieved with a pipeline. Transporting by truck, train, and ship are also possible.
Inject	Depositing CO_2 into the storage reservoir. Potential reservoirs include the deep ocean, ocean sediments, or mineralization (conversion of CO_2 to minerals). Commercial use of CO_2 may also be possible.
Monitor	Once the CO_2 is in the ground, it must be monitored. The main purpose of monitoring is to make sure that the sequestration operation is effective, meaning that almost all the CO_2 stays out of the atmosphere.

The main application of CO_2 capture is likely to be at large point sources such as fossil fuel power plants, fuel processing plants and other industrial plants, particularly for the manufacture of iron, steel, cement and bulk chemicals (Metz et al., 2009). Present available technologies in large power generation plant for separating a concentrated CO_2 stream require both significant additional equipment and a significantly increased input of energy (Irons et al., 2007). The cost of CO_2 capture processes are highly influenced by the design and operation of the CO_2 capture technology applied and also depends on the technical, economic and financial factors related to the design and operation of the production process or power system of interest (Metz et al., 2009).

There are three main CO_2 capture systems associated with different combustion processes, namely, pre-combustion, oxyfuel combustion and post-combustion (Markewitz and Bongartz, 2015). Due to its feasible approach and ease of retrofit to the existing process without much modification, post-combustion capture is the most widely implemented technology for CO_2 capture from emission source (Lee et al., 2012). Post-combustion system can be applied for both coal and gas fired plants (Leung et al., 2014).

1.2 Cassava Leaves as Potential Adsorbent for Carbon Dioxide Capture

Adsorbent produced from biomass waste can help to reduce solid waste and it can be generated into low-cost adsorbent for pollutant removal. In this regard, cassava leaves is used as nitrogen-containing precursor as it is feasible, a biomass derivatives, naturally contain nitrogen found in protein and can be easily obtained.

As nitrogen can be found in amino acids that make up protein, the usage of cassava leaves as raw material for carbon dioxide capture depends on its protein composition. The leaves of 11 varieties of cassava found in Malaysia was harvested from l0 months above old plants and it was found out that the crude protein content ranged from 16.2% to as high as 27.4 % (Devendra, 1978). The potential yield of cassava leaves varies considerably depending on cultivar, age of plant, plant density, soil fertility, harvesting frequency and climate (Ravindran, 1992).

Cassava (Manihot esculenta) is a crop which may support food supply in low rainfall and marginal soil areas which is mostly grown for its roots whereas leaves are typically considered as a by-product and it has become a preferred crop as it needs low input of time, labor and money for both small scale farmers and large-scale plantation (Latif and Müller, 2015). Figure 1.1 shows the hectares of cassava plantation in Southeast Asia.

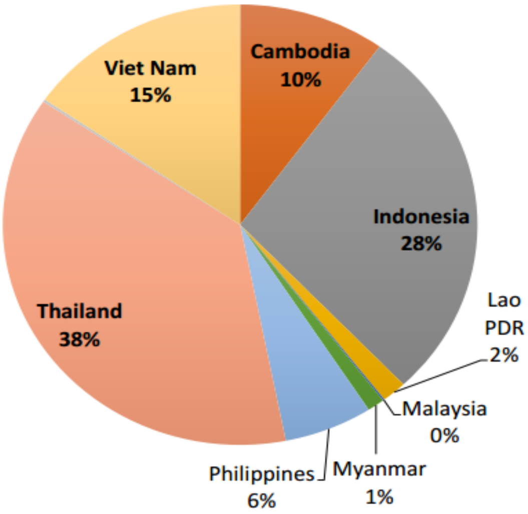

Figure 1.1: Share of Southeast Asia's 3.6 million hectares of cassava (Tan, 2015)

Even though cassava is widely grown and readily obtainable but there are many countries where cassava leaves are not generally consumed and in some countries cassava leaves are only eaten when other vegetable leaves are unavailable or during food shortage and even considered as a poor man's food (Latif and Müller, 2015). Cassava leaves were also used as animal feed and ruminants (Ravindran, 1993; Devendra, 1978).

1.3 Problem Statement

CO_2 is the product of combustion of fossil fuels such as coal. Burning these fuels give out energy which is commonly turned into heat, electricity or power for transportation. As the world population keep increasing, the demand of energy to afford these growth would also increasing. In order to cater the energy demand, more fuels will be needed to combust which in turn releasing more CO_2 to the atmosphere. CO_2 is one of the greenhouse gases that increase the earth temperature that lead to global warming. A method to capture the CO_2 must be implemented.

The increasing carbon dioxide release to the ambient can be controlled by the post-combustion CO_2 capture which trap the CO_2 from the flue gas after the combustion before it was released to the atmosphere. One of the post-combustion CO_2 capture technique include the solid adsorption where a porous carbon was used to capture the CO_2.

Adsorbent used must be carbon that have high surface area, high porosity and has high adsorption capacity to be a good adsorbent for CO_2 capture. It would be more beneficial if the adsorbent can be derived from biomass waste as it can help in reducing the solid waste, low cost and easily obtainable. In this case, a porous carbon derived from cassava leaves was used as an adsorbent to capture the CO_2 gases. Cassava leaves can considered as a good adsorbent as it is considered as biomass derivatives, easily obtainable and is low cost. In addition, cassava leaves contain an amount of nitrogen in the form of protein than can enhance the performance for CO_2 capture. The intention of this research is to study the performance of a naturally contained nitrogen compound as the raw material.

1.4 Research Objectives

The objectives of this research are to:

1. Prepare adsorbents derived from cassava leaves for carbon dioxide (CO_2) capture.
2. Characterize the as prepared adsorbents for its chemical and physical properties.
3. Investigate the adsorption performance of the prepared adsorbents for CO_2 capture using different parameters such as CO_2 concentration, adsorption temperature and total inlet flowrate.

1.5 Scope of Study

In this study, cassava leaves was used as an adsorbent for CO_2 capture. It was first dried overnight to remove the moisture content. The dried leaves was crushed before undergo carbonization. Then it was carbonized at 500 °C, 550 °C and 600 °C under nitrogen flow for 90 min. Then adsorption study was carried out to determine the best temperature for carbonization. After optimum temperature for carbonization has been identified, the char produced from carbonization at that temperature will be mixed with KOH (potassium hydroxide) at different ratio. The ratio that will be tested are 1:0.5, 1:1, 1:2 and 1:3 with respect to char/KOH. Then all the prepared sample will undergo activation process at temperature of 800 °C for 90 min and this will produce activated carbon. Adsorption study will once again be carried out to identify the best ratio of char/KOH. Then the finest sample will be used to test different parameter for fixed bed adsorption which are CO_2 concentration, total inlet flowrate and adsorption temperature.

1.6 Thesis Organization

This part thesis consists of five main chapters and each chapter contributes to the sequence of this study. The following are the contents for each chapter in this study:

Chapter One - introduce the overview of this research on CO_2 capture, problem statements, research objectives, research scope and the organization of this thesis.

Chapter Two - discusses the literature review of this study. This chapter briefly presents the previous findings, reviews and references available that are related to this research topic. In general, this chapter outlines the overview and elaboration on post-combustion CO_2 capture using solid adsorbent derived from biomass derivatives. Then, an insight of post-combustion CO_2 capture, adsorption using solid adsorbents, activated carbon and biomass waste as source for adsorbent preparation were provided.

Chapter Three - covers the experimental materials and methods used in this research. It presents the materials, chemicals and gases required along with the description of equipment and the adsorbent preparations. Then, a description of the experimental methodology along with the adsorbent characterization method used in this research project is presented.

Chapter Four – refers to the experimental results and discussions of the data obtained. Provide data and result on adsorbent preparation parameter (carbonization temperature and chemical activation char/KOH ratio) and parameters for fixed bed adsorption (CO_2 concentration, total inlet flowrate and adsorption temperature). Further elaboration on the adsorbent breakthrough studies, adsorption capacity, characterization results and the effects of different factors on fixed bed adsorption are provided in this chapter. Then the characteristics of the adsorbent were also studied.

Chapter Five - concludes all the findings achieved in this research study. Recommendations for future studies on this research topic are included as well.

CHAPTER TWO

LITERATURE REVIEW

This chapter discusses the literature review of this study. This chapter briefly presents the previous findings, reviews and references available that are related to this research topic. In general, this chapter outlines the overview and elaboration on post-combustion CO_2 capture using solid adsorbent derived from biomass derivatives. Then, an insight of post-combustion CO_2 capture, adsorption using solid adsorbents, activated carbon and biomass waste as source for adsorbent preparation were provided.

2.1 Post-combustion Carbon Dioxide Capture

CO_2 is a product of combustion and the appropriate carbon dioxide removal process depends on the type of the combustion. In order to decrease CO_2 emissions, new-designed power plants and renewable energies have been established to lessen or eliminate these CO_2 emission. Figure 2.1 shows the main CO_2 contributor.

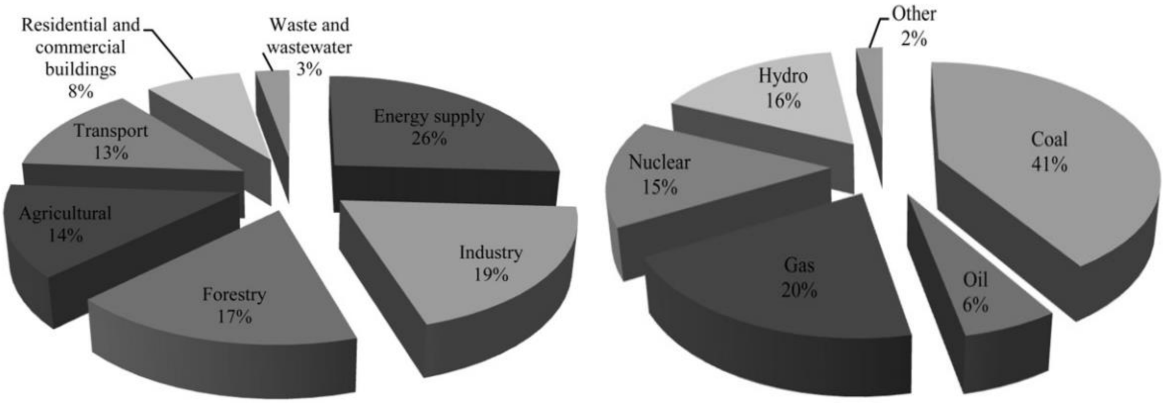

Figure 2.1: Main CO_2 contributor (Lee et al., 2012)

From figure 2.1, it can be seen that the energy supply is the second main contributor with 28% towards CO_2 emissions. As energy supply is related to combustion of fossil fuels, necessary action need to be taken in order to reduce the CO_2 emissions to the atmosphere.

Post-combustion capture is the removal of carbon dioxide from the flue gas after the combustion (Blomen et al., 2009) and the techniques of post-combustion mainly involve chemical absorption, physical adsorption, membrane separation and cryogenic separation (Lee et al., 2012).

As separation by membranes are under development and do not yet exist for an industrial scale (Kanniche and Bouallou, 2007) and cryogenic separation requires high energy that resulting for higher operating cost, chemical absorption and physical adsorption gain more focus compared to cryogenic separation and membrane separation (Oh, 2010). By far, chemical absorption with amine-based or ammonia-based adsorbent is widely performed by industrialists as it receives the greatest attention due to its high process efficiency (Rashidi and Yusup, 2016).

In post-combustion capture, amine scrubbing is favourable as it has been commercially proven on a small scale and it can be retrofitted to existing power plants and industries in suitable locations (Blomen et al., 2009). However, the utilization of chemical absorption had raised concerns due to amine degradation, either through oxidative degradation which cause the amine compounds to fragment into toxic derivatives (organic acid, ammonia, amide) or through thermal degradation that produce large molecular weight of the amine-chain product and there is a possibility that a minor portion of these chemicals will evaporated and released to the atmosphere together with the clean flue gas due to the high volatility of these amine compounds (Rashidi and Yusup, 2016).

Solid adsorption process is recommended as an alternative to the present technology due to the shortcomings of conventional amine scrubbing process in carbon dioxide removal from flue gas. Adsorption systems are simple in design and easy to operate (Eze and Agbo, 2010). It has good moisture removal capacities and the process can remove CO_2, H_2S, moisture and other impurities either selectively or simultaneously (Kapdi et al., 2005). Figure 2.2 shows the basic flow diagram for CO_2 capture via adsorption.

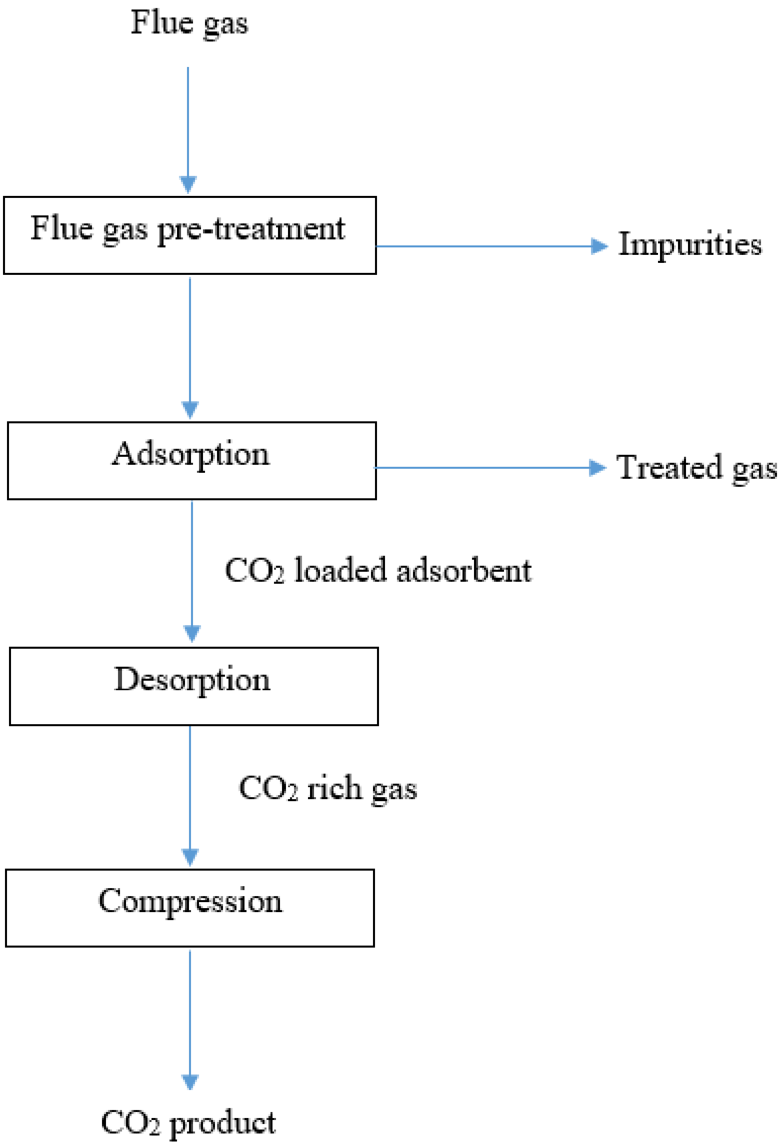

Figure 2.2: Basic flow diagram for CO_2 capture via adsorption (Spigarelli and Kawatra, 2013)

There are a few guiding principle that must be taken by researchers in considering the adsorption technology as an alternative to amine scrubbing process so that effectual separation process is achieved. Accordingly, the selection of adsorbent should satisfy the following features which include high adsorption capacity, high surface area, fast kinetic, high CO_2 selectivity and mild regeneration conditions (Spigarelli and Kawatra, 2013). Favourable adsorbents should also need to have a high stable adsorption-desorption cycles, tolerance to moisture and impurities, and low energy requirements (Li and Hitch, 2015).

2.2 Adsorption using Solid Adsorbents

Among existing technologies for CO_2 capture, adsorption using solid porous adsorbents is regarded as the most favourable method. Adsorption mainly depends on the development of new adsorbent with excellent pore characteristics, adsorption capacity, selectivity, and fast kinetics (Yaumi et al., 2017). There are several types of solid adsorbents being considered as potential CO_2 capture materials such as molecular sieves, porous silica, metal–organic frameworks, membranes and porous carbon (Liu et al., 2013). Table 2.1 lists and compares advantages and disadvantages of some typical solid adsorbents for CO_2 capture.

Carbon-based materials is an outstanding materials for CO_2 adsorption due to the high CO_2 adsorption rates, good selectivity for CO_2-N_2 separation, easily regenerated, easy-to-design pore structure and a wide availability of sources for carbons which makes industrial-scale production relatively low-cost (Sevilla and Fuertes, 2012; Choi et al., 2009; Sevilla et al., 2011). Porous carbon is an ideal solid adsorbent among others also due to its large surface area, high porosity and good chemical and physical stability (Liu et al., 2013). Figure 2.3 display the different type of porosity that are present in an adsorbent.

Table 2.1: Comparison between major non-carbonaceous adsorbents for CO_2 capture (Lee and Park, 2015)

Adsorbents	Advantages	Disadvantages
Zeolites, silica materials	• Low production cost • Large micropores and mesopores • Medium CO_2 adsorption (at 298 K and 1 bar)	• Poor performance of CO_2 adsorption due to easy moisture absorption • Heavy energy consumption during CO_2 desorption (poor economic feasibility) • Renewal difficulties
Alkali-based dry adsorbents	• Possible adsorption and desorption at a low temperature, i.e., 313-343 K (similar to amine-based absorption) • Possible CO_2 collection under wet conditions • Absorption and renewal under 473 K and possible operation at atmospheric pressure (high economic efficiency)	• Low adsorption capability (3–11 wt. %) • High-temperature reactions • Decrease in the collection ratio of CO_2 because of stable products • Requires high temperatures during desorption (high energy consumption) • Complicated operation

Table 2.1: Continued

Metal organic frameworks (MOFs)	• Large specific surface area (over 10,000 m^2/g) and regular pore distributions • Ease of controlling pore sizes • Possible improvement in CO_2 selectivity according to various combinations of metal clusters and organic ligands	• Poor performance at the partial pressure of CO_2 • Poor economic efficiency due to high production cost • Complicated synthetic process • Possible structure failure due to moisture absorption during CO_2 capture • Unsuitable for use at high temperature.
Metal oxides based adsorbents	• Dry chemical absorbents • Adsorption/desorption at medium to high temperatures (>673 K) • Popular as a pre-combustion absorbent	• High consumption of energy due to adsorption/desorption at medium to high temperatures (>673 K) • High cost for regeneration • Demand for continuous addition of absorbents • Complicated process

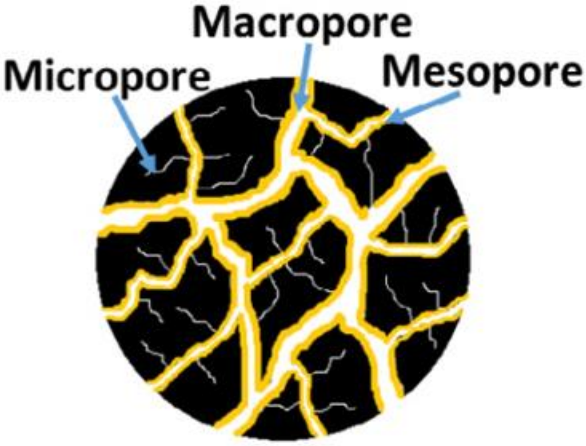

Figure 2.3: Graphical illustration for iron oxide coated granular activated carbon by Suresh et al., (2017)

2.3 Activated Carbon

Activated carbon are carbon that have high surface area and porosity and it can be used as an absorber in the industrial sector to reduce the pollution that come from the effluent in the form of water or air. It is a processed carbon material with a large internal specific surface area and highly developed porous structure (Abdullah et al., 2001). As the major challenges associated with the usage of activated carbon for adsorption is cost-effectiveness, researchers are more focused of developing an alternative for the commercialized production of activated carbon from agricultural waste materials (Sivakumar et al., 2012). The material used to produce activated carbon can be any material with a high amount of carbon content. A variety of carbonaceous material such as wood, coal, lignite and coconut shell can be used to produce activated carbon (Bhatnagar et al., 2013). Figure 2.4 shows the SEM image of activated carbon from sawdust of Balsamodendron caudatum wood waste with activation at 800°C for 60 min (Sivakumar et al., 2012).

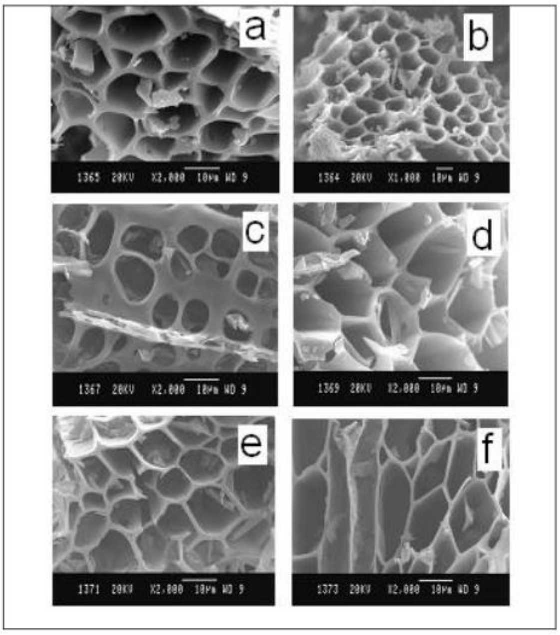

Figure 2.4: SEM image of activated carbon (Sivakumar et al., 2012)

Activated carbon is a useful material as its surface has a wide spectrum of functional groups present along with a high surface area, large porosity and a well-developed internal pore structure consisting of micro-, meso- and macropores making it valuable in various applications in many areas, but mainly in the environmental field (Bhatnagar et al., 2013). An outstanding surface properties and specific functionalities with a sufficient amount of super microporosity and mesoporosity can create a high affinity for the adsorption of adsorbate and will be beneficial for the improved solute adsorption (Sivakumar et al., 2012). An activated carbon with well-developed pore shape and porosity can be produce by carbonization and subsequent chemical activation of nitrogen-containing precursors derived from biomass under inert atmosphere (Yaumi et al., 2017).

2.3.1 Activated Carbon Preparation

Activated carbon can be prepared by using physical or chemical activation process. Both process can produce activated carbon with different pore sizes and the development of porosity is different in term of practical procedures and mechanism (Abdullah et al., 2001). Apart from the nature of the precursors, the operating condition also play an important role in developing suitable activated carbon for gas phase application. There are two type of activation in activated carbon production which are physical and chemical activation.

In chemical activation, activated agent is reacted with the carbon at high temperatures to remove the majority of hydrogen and oxygen from the carbon structure and it often combines the carbonization and activation step, but these two steps may still occur separately depending on the process. Some of the activation agents that are usually used include alkaline, acidic, and salt mediums such as potassium hydroxide (KOH), sodium hydroxide (NaOH), zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4) (Rashidi and Yusup, 2016). In one step chemical activation process, the precursor is mixed with a chemical such as $ZnCl_2$ or H_3PO_4, carbonized and washed to produce the activated carbon (Abdullah et al., 2001) while in two step, the precursor will first carbonized before it was impregnated with activator and then undergo activation. Table 2.2 shows the summary of previous works of preparation of activated carbon produced by different raw materials by using chemical activation.

Table 2.2: Summary of CO_2 adsorption onto different activated carbons

Precursor	System	Carbonization		Activation					Max Surface Area (m^2/g)	Adsorption Capacity for CO_2 (mmol/g)			References
		Temp (°C)	Holding Time (min)	Types of oxidant	IR[a] (wt/wt)	Temp (°C)	Holding Time (min)	Heating Rate (°C/min)		0 °C	25 °C	70 °C	
Coconut Shell	Continuous	600	n/a	KOH	1:3	800	120	10	985	-	3.5[b]	1.70	(Yaumi et al., 2017)
Palm Shell	n/a	600	60	KOH	1:3	850	60	10	1250	6.3	4.4	-	(Ello et al., 2013)
Peanut Shell	n/a	500	90	KOH	1:1	700	90	5	956	5.2	4.0	-	(Deng et al., 2015)
Celtuce Leaf	n/a	600	n/a	KOH	1:4	800	60	n/a	3404	6.0	4.4	-	(Wang et al., 2012)

[a] IR = Impregnation Ratio. (Char/Activator)

[b] CO_2 Adsorption at 30°C.

2.3.2 Surface Modification

There are many works are based on improving the adsorptive properties of porous adsorbents. By modifying their surfaces, the basic feature can be improved. Such alterations include exchange or substitution of cations within the metal framework, chemical treatment of the surface or functionalization of the pores and the modifications usually result in a decrease of the surface area but increase the adsorptive selectivity and capacity for CO_2 (Langlois et al., 2016). Surface alteration of these carbon-based adsorbents can promote their affinity and selectivity towards the CO_2 molecule.

In recent years, some basic functional groups have been introduced intentionally to porous carbons to promote their CO_2 adsorption capacity and selectivity as previous studies have shown that the CO_2 uptake on pure porous carbons is not adequate (Liu et al., 2013). To create a superficial basic sites through the incorporation of nitrogen groups into the carbon framework, a large amount of research effort has been engaged in order to improve the CO_2 adsorption capacity of porous carbons. The mechanical properties and surface chemistry can be also improved by the incorporation of nitrogen atoms in the carbon nanostructure (Lu et al., 2014).

Some nitrogen-containing carbon materials have been used as precursors, for instance polyacrylonitriles, acetonitrile, polyvinylpyridine, or pyrrole, but they are less viable and available compared to biomass derivatives (Yaumi et al., 2017). Figure 2.5 shows the FTIR image of nitrogen-doped activated carbon from raw coconut shell mixed with glucosamine that represent various functional group found in the precursor.

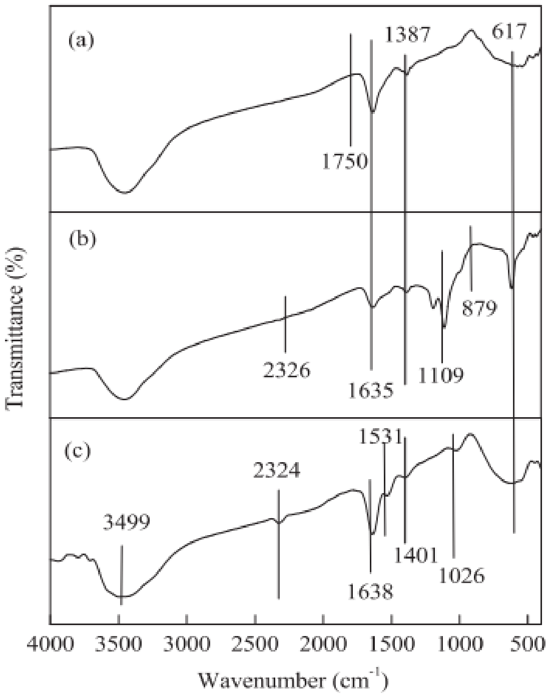

Figure 2.5: FTIR image of activated carbon derived from coconut shell (Yaumi et al., 2017)

2.4 Summary

In this chapter, CO_2 capture technologies had been discussed. The CO_2 capture using solid adsorption was found to be a potential precursor to capture CO_2. The challenge is to select the precursor from a sustainable resources such as biomass residue which is easily obtainable. In this study cassava leaves was selected as the precursor as it is a form of biomass waste and it can be easily available. The study of operating conditions in adsorption process also had been reported and discussed in this chapter. The effect of the ratio of activation agent to char, total gas flowrate, adsorption temperature and initial CO_2 concentration on the adsorption performance will also be studied in this work.

CHAPTER THREE

MATERIALS AND METHODS

This chapter describes the experimental procedures that need to be carried out including the materials, chemicals and equipment used while conducting this research. The overall experimental works can be divided into four sections which are preparation of char from precursor (cassava leaves) by carbonization, preparation of activated carbon from char using chemical activation, carbon dioxide capture study and the characterization study.

3.1 Research Activities

Flow Diagram of research activities is shown in Figure 3.1.

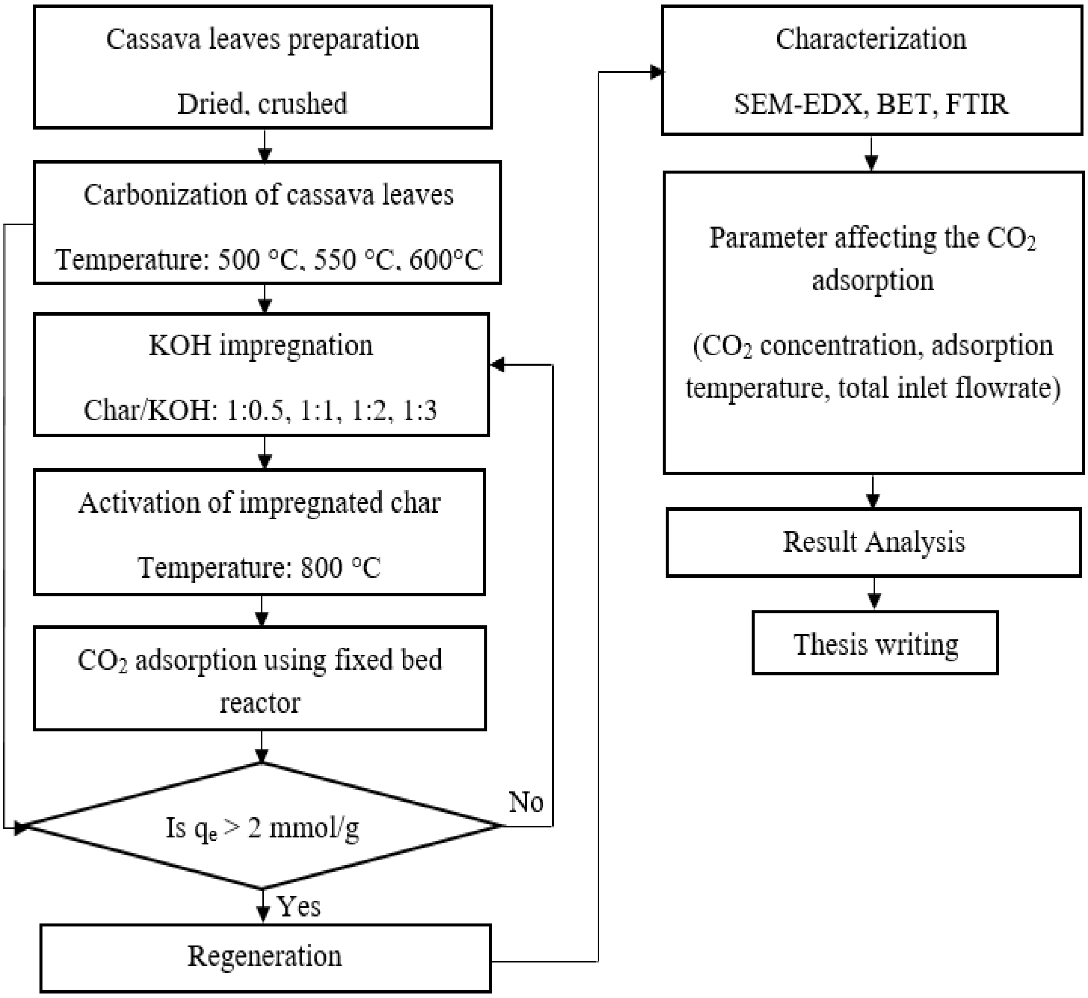

Figure 3.1: Flow diagram of the research activities

3.2 Materials and Chemicals

3.2.1 Material

In this study, cassava leaves was used as the raw material to produce activated carbon. It was collected from Kampung Simpang Temerlok, Perak. Figure 3.2 shows the cassava leaves used in this experiment.

Figure 3.2: Cassava leaves

3.2.2 Chemical and Gases

The chemical used in this research are listed in Table 3.1.

Table 3.1: List of chemical and gases

Chemicals	Supplier	Purity, %	Purpose of use
Potassium Hydroxide (KOH)	Merck, Germany	99.9	Activating agent
Nitrogen Gas (N_2)	Wellgas Sdn. Bhd. Malaysia	99.9	Create inert atmosphere for carbonization and activation process
Carbon Dioxide Gas (CO_2)	Wellgas Sdn. Bhd. Malaysia	99.9	Flue gas in adsorption study

3.3 Equipment Description

3.3.1 Horizontal Tubular Reactor

Figure 3.3 shows the horizontal tubular reactor used in this experiment while Figure 3.4 shows the schematic diagram of the horizontal tubular reactor.

Figure 3.3: Horizontal tubular reactor

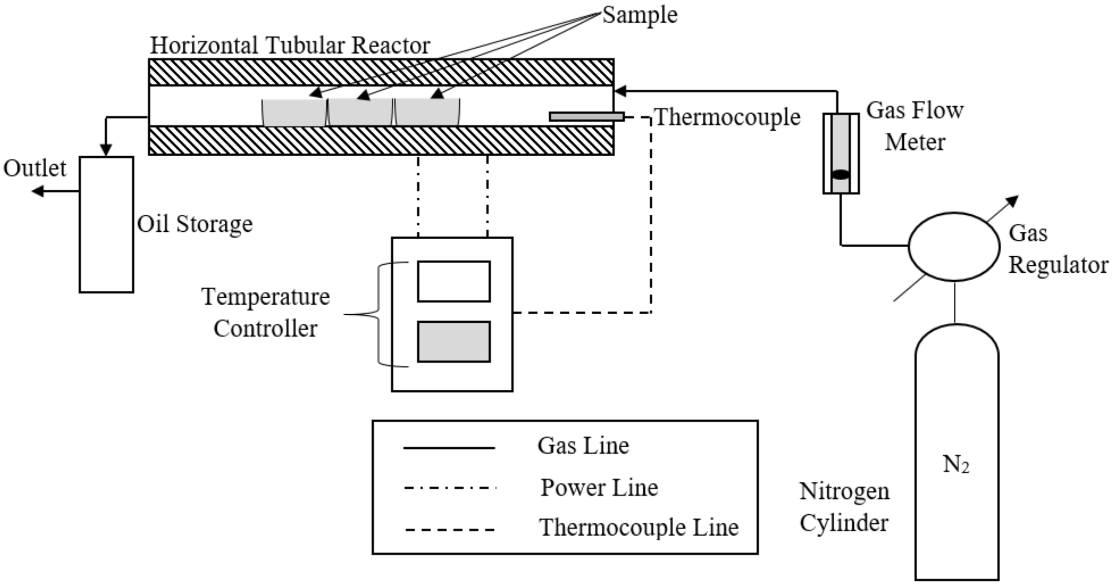

Figure 3.4: Schematic diagram of horizontal tubular reactor

Horizontal tubular reactor was used to carbonize dried cassava leaves to obtain char and to activate the impregnated char to obtain activated carbon. The sample was put in a crucible and the crucible was inserted into the reactor. The N_2 gas flowing inside the reactor was controlled by the gas flow meter. The temperature of the reactor was controlled by the temperature controller.

3.3.2 Fixed Bed Adsorption System

Figure 3.5 shows the complete setup of fixed bed adsorption system used in this experiment. Figure 3.6 shows the schematic diagram of fixed bed adsorption system used in this experiment.

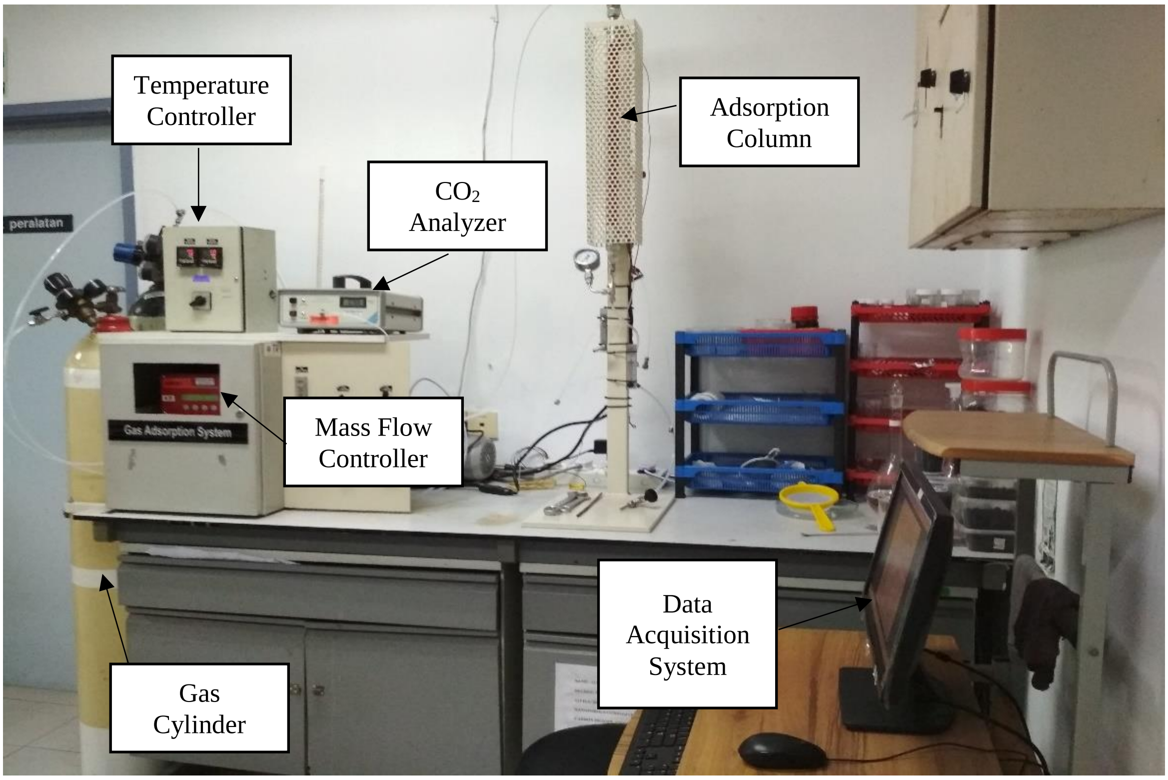

Figure 3.5: Complete setup for fixed bed adsorption system

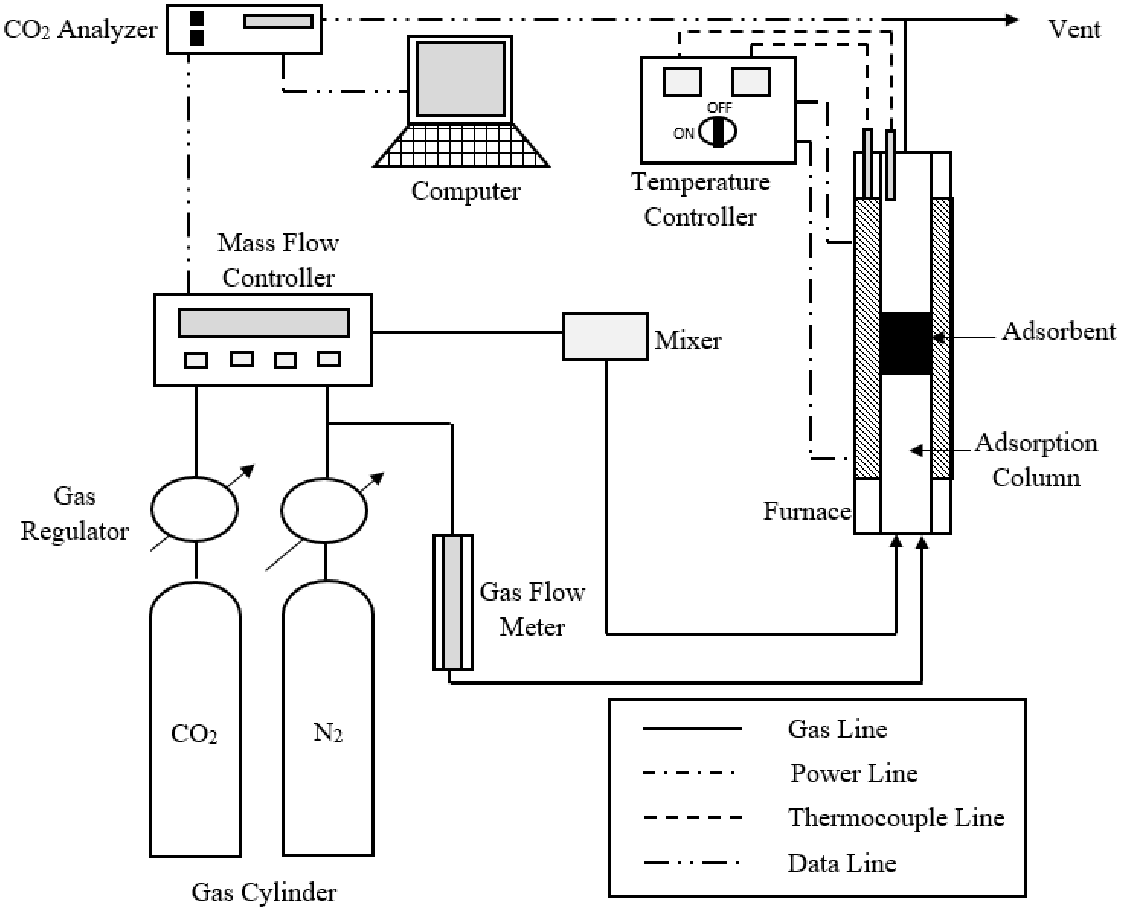

Figure 3.6: Schematic diagram of fixed bed adsorption system

Fixed bed column was used to study the adsorption of carbon dioxide onto the activated carbon produced. In this system, CO_2 and N_2 gas flowing into the system will be controlled using the mass flow controller (AFC26, Aalborg Instrument and Control, Inc., Germany). The column was surrounded by heating element and the length of the column is 14 cm with inner diameter of 1.1 cm. The temperature controller was used to control the column temperature to the desired adsorption temperature. The amount of CO_2 adsorbed was measured through an online CO_2 analyzer (model 906, Quantek Instrument, Inc., Grafton, MA) connected to computer for online data logging. The amount of CO_2 adsorbed was monitored online every 10 seconds. The amount adsorbed was calculated from the breakthrough curves generated by the CO_2 analyzer.