Production of Sea Mango (*Cerbera Odollam)* Based Activated Carbon for CO_2 Adsorption

by

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A thesis submitted in fulfillment of the requirements for the degree of
Master of Science in Environmental Engineering

School of Environmental Engineering
UNIVERSITI MALAYSIA PERLIS

2016

ACKNOWLEDGEMENT

Alhamdulillah, praise to Allah S.W.T for His blessing and strength given to me for completing this research and thesis. This research would have not been accomplished without the support, help and guidance of a number of people. First, I would like express my gratitude especially to my supervisor, Dr. Umi Fazara Md. Ali, for accepting me as her student, the guidance, encouragement and continuous supervision as well as providing necessary equipment and financial assistant associated with this research. Next, I would like to express my special gratitude and thanks to Prof. Dr. Mohamed Kheireddine Aroua for the guidance, advice and financial support in completing the research. A special thanks goes to my co-supervisor Miss Nor Fauziah Zainudin for all the assistant and advice given. I would also like to thank Kementerian Pengajian Tinggi (KPT) for the Research Acculturation Collaborative Effort (RACE 9017-00017) grant, MyMaster under the Mybrain15 program as well as grant from Rakan Penyelidikan UM.

Lastly, my greatest gratitude goes to my family and family in law, especially to my lovely husband Mohd Yazrizal Ibrahim and my dearest daughter Nur Laura Kasturi Mohd Yazrizal. Without their blessing, I would not be able to be where I am today. I love you all. Finally thanks for all the support and encouragement given to me from those who involved directly or not during my research and also to my friends that give me their highest supports and advice. Thank You.

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LIST OF ABBREVIATIONS

AC	Activated Carbon
CO_2	Carbon Dioxide
N_2	Nitrogen
AMP	2-Amino-2-Methyl-1-Propanol
PZ	Piperazine
DEA	Diethanolamine
MEA	Ethanolamine
MDEA	N-Methyldiethanolamine
UNFCC	United Nations Framework Convention on Climate Change
COP	Conference of the Parties
CCS	Carbon Capture and Storage
BET	Brunauer–Emmett–Teller
SEM	Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
KOH	Potassium Hydroxide
$NaCO_3$	Sodium Carbonate
K_2CO_3	Potassium Carbonate
IR	Impregnation Ratio
H_3PO_4	Phosphoric Acid
$ZnCl_2$	Zinc Chloride
ppm	Parts Per million

HCl	Hydrochloric Acid
RCIHM2-A	Activated Carbon Prepared from Method 2 with Absence of any Gases
RIHM1-N	Activated Carbon Prepared from Method 1 with Nitrogen Gases
C	Carbon Element
H	Hydrogen Element
N	Nitrogen Element
O	Oxygen Element
S	Sulfur Element
DP	Double Porosity
MW	Molecular Weight
kBr	Potassium Bromide

LIST OF SYMBOLS

°C	Celcius
wt. %	Percentage By Weight
S_{BET}	Specific Surface Area BET
atm	Standard Atmosphere
K	Kelvin
V_{mic}	Micropore Volume
V_{meso}	Mesopore Volume
V_T	Total Pore Volume
nm	Nanometer
P/P_o	Relative Pressure
mL/min	Milliliter Per Minute
C/C_o	Relative Concentration of CO_2 Leaving the Adsorption Bed
$mg_{CO2}/g_{sorbent}$	Milligram of CO_2 per 1 gram sorbent
L	Avogadro constant
σ	Cross-Sectional Area of an Adsorbate
V_m	Amount Monolayer Gas Adsorbed
N	Normality
L/m	Liter per Meter
t_b	Breakthrough Time
s^{-1}	1 per second
cm/s	Centimeter Per Second

mm	Millimeter
rpm	Rotation Per Minute
$m^3/s/kmol$	Meter Cube Per Second Per Kilo Mole

Penghasilan Mangga Laut (*Cerbera Odollam*) yang Berasaskan Karbon Teraktif bagi Penjerapan CO_2

ABSTRAK

Penggunaan karbon teraktif bagi proses penjerapan karbon dioksida (CO_2) merupakan kaedah terbaik bagi mengurangkan pelepasan gas karbon dioksida. Karbon teraktif telah dihasilkan daripada buah yang tidak boleh dimakan mangga laut (*Cerbera odollam*) melalui penggabungan proses kimia dan fizikal. Terdapat dua kaedah penyediaan karbon teraktif; iaitu Kaedah 1: bahan mentah telah direndam dengan asik fosforik (H_3PO_4) dan kemudiannya diaktifkan pada suhu 500 °C dengan pengaktifan atmosfera yang berbeza selama 2 jam, Kaedah 2: bahan mentah telah dipanaskan pada suhu 200 °C dengan menggunakan aliran gas nitrogen (N_2) terlebih dahulu sebelum proses pengaktifan kimia dan haba seperti di Kaedah 1. Struktur pori wujud pada permukaan karbon teraktif hasil daripada proses pengaktifan fizikokimia dan telah dibuktikan oleh kajian morfologi permukaan. Karbon teraktif bersih yang terbaik telah dihasilkan oleh pengaktifan dua-peringkat melalui Kaedah 2 dalam ketidakhadiran sebarang gas (RCIHM2-A) yang mana telah mencatatkan keluasan spesifik BET (1475.43 m^2/g) tertinggi, kapasiti penjerapan iodin (1040.58 mg/g) dan elemen karbon tertinggi (79.17 %). Proses pengaktifan yang berlaku kemudiannya disahkan dengan ketiadaan perubahan dan/atau kehilangan kumpulan berfungsi C-H dan -C≡C-H:C-H pada puncak 2936.73 dan 617.76 cm^{-1} berdasarkan perbandingan dengan bahan mentah mangga laut. Keterbatasan karbon teraktif yang bersifat pori meso dengan pori berdiameter 2.86 nm kemudiannya diubahsuai dengan rendaman amina berasaskan kimia (AMP, PZ, MEA, dan DEA) untuk mempertingkatkan keupayaan penjerapan semulajadi dan pemilihan bagi menjerap CO_2. Proses penjerapan fizikal yang mana mempunyai interaksi daya van der waals yang lemah diantara molekul CO_2 dan pori pada permukaan karbon teraktif bersih telah diubah kepada proses penjerapan kimia (ikatan kovalen) melalui perendaman amina. Prestasi karbon teraktif terhadap penjerapan CO_2 telah diperolehi melalui lengkung bulus dan kapasiti penjerapan. Pengurangan keluasan permukaan BET sebanyak 56 % hingga 62 % selepas perendaman amina membuktikan molekul amina telah berjaya ditempatkan di atas permukaan karbon teraktif. Keputusan kemudiannya telah disahkan oleh imej SEM yang mana menggambarkan molekul amina telah memenuhi keseluruhan pori karbon teraktif. Amina terbaik yang telah difungsionalisasikan di atas permukaan karbon teraktif ialah 2-amino-2-methyl-1-propanol (AMP) dengan kapasiti penjerapan CO_2 tertinggi sebanyak 23.05 $mg_{co2}/g_{penjerap}$ dan mampu mencapai 94.47 % penjanaan semula. Pengukuran elemen nitrogen (N) melalui analisa elemen dengan menggunakan EDX meningkat sebanyak dua kali ganda (daripada 4.73 % kepada 7.25 %) selepas dengan rendaman dengan amina AMP ini dan telah disahkan dengan kewujudan ikatan N-H di 3391.22 cm^{-1} dan ikatan C-N masing-masing di puncak 1173.19 dan 1047.55 cm^{-1}. Keputusan menunjukkan elemen N telah mengfungsionalisasikan permukaan karbon teraktif bagi penjerapan CO_2. Kesan bagi parameter lain seperti aliran masuk gas (5.00 mL/min) dan kuantiti sampel (2.00 g) terendah mencatatkan kadar penjerapan tertinggi iaitu masing-masing 23.05 dan 28.18 $mg_{co2}/g_{penjerap,}$ manakala, penjerapan CO_2 adalah berkadar songsang dengan peningkatan aliran masuk gas dan kuantiti sampel.

Production of Sea Mango (*Cerbera Odollam)* Based Activated Carbon for CO_2 Adsorption

ABSTRACT

The activated carbon utilization for carbon dioxide (CO_2) adsorption process is a promising method to reduce the emission of CO_2. The activated carbon was produced from the non-edible sea mango (*Cerbera odollam*) fruit through sequential chemical and physical processes. There were two methods of activated carbon preparation; i.e. Method 1: the precursor was impregnated with phosphoric acid (H_3PO_4) and then thermally activated at 500 °C with different activation atmosphere for 2 hours, Method 2: the precursor was heated at 200 °C with nitrogen gas (N_2) flow prior to chemical and thermal activation processes as performed in Method 1. Porous structures were developed on the surface of the activated carbon due to the physicochemical activation process and proven by surface morphology study. The best virgin activated carbon prepared by double-stage activation via Method 2 in the absence of any gases (RCIHM2-A) which recorded the highest BET surface area (1475.43 m^2/g), iodine adsorption capacity (1040.58 mg/g) and the highest carbon element (79.17 %). The activation process was further confirmed to happen with the unchanged and/or disappearance of C-H and -C≡C-H:C-H of the functional group at peaks 2936.73 cm^{-1} and 617.76 cm^{-1} as compared to raw sea mango sample. The prepared mesoporous activated carbon with pore diameter 2.86 nm was then modified with amine-based chemical (AMP, PZ, MEA, and DEA) impregnation to improve its adsorption capability and selectivity to adsorb CO_2. Physisorption process which has weak Van der Waals forces interaction between CO_2 molecules and pores on virgin activated carbon surface has been transformed to chemisorptions process (covalent bond) *via* amine impregnation. The performance of the activated carbon on CO_2 adsorption was obtained through breakthrough time curve and adsorption capacity. The reduction of BET surface area was approximately 56 % to 62 % after amine impregnation proved that amine molecules successfully attach on the activated carbon surface. The result was further confirmed by SEM images which illustrated that amine molecules filled up most of the activated carbon pores. The best amine functionalized on carbon surface was 2-amino-2-methyl-1-propanol (AMP) with highest CO_2 adsorption capacity 23.05 $mg_{CO2}/g_{sorbent}$ and has been able to achieve 94.47 % of regeneration. The nitrogen (N) element measurement via elemental analysis by EDX was enhanced two-fold (from 4.73% to 7.25 %) after impregnated with this AMP amine and confirmed by the existence of N-H bond at 3391.22 cm^{-1} and C-N bond at 1173.19 and 1047.55 cm^{-1}, respectively. The result indicated that elemental N functionalized activated carbon surface for CO_2 adsorption. The effect of other operating parameters such as the lowest inlet flow rate (5.00 mL/min) and sample loading (2.00 g) recorded the highest CO_2 adsorption capacity, 23.05 $mg_{CO2}/g_{sorbent}$, and 28.18 $mg_{CO2}/g_{sorbent}$, respectively, whilst CO_2 adsorption capacity was inversely proportional to inlet flow rate and adsorbent loading increment.

CHAPTER 1

INTRODUCTION

1.1 Introduction

Currently, there are growing concerns on the environmental issues particularly on global warming due to the anthropogenic release of carbon dioxide (CO_2) to the environment. Such uncontrollable CO_2 release from human activities, e.g. burning of fossil fuels, have been identified to be the major contributor to the climate change that requires sustainable technological solutions to lessen their impact on the environment.

In this respect, the Carbon Capture and Storage (CCS) concept has been introduced recently as part of CO_2 mitigation strategies predominantly consist of two sequential steps i.e. trapping CO_2 followed by storing it in a sustainable manner. In the former step, the CO_2 can be effectively captured from gas mixtures using conventional gas separation techniques such as gas-liquid absorption and gas-solid adsorption. Alternatively, membrane based separation techniques offer more advanced features over the conventional counterparts in view of the size and its operational characteristics. In the later step, the CO_2 captured can be chemically converted to a stabilized form of metal carbonate for safe disposal or it can be readily transformed into chemicals of keen interest via chemical or biological routes.

A number of technologies have been around for CO_2 capture such as cryogenic separations (Tuinier, et al., 2010), gas absorption (Xu, et al., 2014), membrane separations (Brunetti, et al., 2010), calcium looping (Dieter, et al., 2014) and adsorption (Olivares-Marín, et al., 2011). Although various chemical and physical techniques for CO_2 capture have been established, adsorption using activated carbon (AC) is a well-known method since the material provides a large surface area, high porosity, stable in acidic or basic solutions, and it is cost effective as AC can be regenerated (Lee, et al., 2013). Moreover, according to Houshmand et al. (2012), the surface of the AC can be modified to enhance the adsorption capacity.

There are various surface modification techniques available to improve the adsorption capacity of ACs including heat treatment, oxidation, chemical treatment with metallic species, and acidic or basic solutions (Sui, et al., 2007; Sun, et al., 2007; Manocha, 2003). Lee et al. (2013) and Khalil et al. (2012) suggested a chemical impregnation of AC with alkanolamine which will promote nitrogen functional group on the porous adsorbent surface. The research discovered that the CO_2 adsorption capacity improved by two-fold with percentage improvement (19 to 73 %) and (89 to 172 %) for Lee and Khalil's investigation, respectively.

1.2 Research Motivation

The combustion of fossil fuels especially for energy generation has produced significant quantities of CO_2 which is one of the main causes of global warming. During the 2009 United Nations Framework Convention on Climate Change (UNFCCC) Conference held in Copenhagen, Prime Minister, Dato' Seri Mohd Najib Tun Abdul Razak announced that Malaysia would aim to reduce its CO_2 emissions intensity per gross domestic product (GDP) by 40 % by the year 2020 compared to 2005 levels which cost at RM 3750 GDP per ton of CO_2 emission. Therefore, in 2010, Malaysia launched its National Policy on Climate Change, which provides an overarching policy framework for achieving Malaysia's CO_2 emissions reduction targets including the application of carbon capture and storage (CCS) technology.

The chemical absorption amine-based or ammonia-based absorption processes has been implemented in petrochemical industries for CO_2 capture. However, there are some drawbacks such as small contact area between gas and liquid, low CO_2 loading, and harmful absorbent corrosion. Regeneration of liquid solvent is also relatively costly because of the high heat capacity and volume of water consumed in the process. Hence, solid adsorption process e.g. using AC is a potentially viable method to capture CO_2.

AC is an adsorbent widely used in solid adsorption process due to its favourable characteristics such as highly porosity, high surface area, high adsorption capacity and cost effectiveness through regeneration. In this study, AC adsorbents are prepared from a novel precursor, sea mango (*Cerbera odollam*) which is normally considered a waste. Conversion of this waste into ACs would add value to this ornamental fruit, reduce the cost of disposal and provide a potentially cheap alternative to existing commercial coal based ACs.

In AC production, type of activating agent play an important role and should take into account to produce good quality of AC. The inferior surface area of the resulting AC produced from KOH, make KOH is not a favourable activating agent in chemical activation. On the other hand, $ZnCl_2$ also not good choice activating agent due to contamination issue by zinc. Thus, H_3PO_4 is the best activating agent due to the superior quality of AC produced with higher surface area and successfully overcome the product contamination issue.

The CO_2 adsorption capacity of AC depends on their surface chemistry and porosity properties. Hence, surface treatment using minerals, chemical or other materials is required to improve its natural capability for gas adsorption. CO_2 adsorption capacity can be improved by promoting nitrogen functional group by chemical impregnation such as amine and ammonia.

The novelty of this research is highlighted with the application of sea mango as AC precursor for CO_2 adsorption process. Although there have been some studies carried out on this fruit for biodiesel production, production of AC from this natural waste is still in its infant stage and its potential use for CO_2 adsorption have not been explored yet. Additionally, the AC that being produced in this research work has its own formulated chemical composition and synthesis steps which requires low temperature, and able to generate quality AC with good adsorption characteristics.

1.3 Objectives

The main objectives of this study are:

i. To prepare and characterize the activated carbon from the non-edible sea mango (*Cerbera odollam*) fruit.

ii. To impregnate the new solid sorbents with alkanolamines for CO_2 capture.

iii. To evaluate the performance of these adsorbent in capturing CO_2 focusing on breakthrough curves.

1.4 Scope of the Study

AC is prepared from non-edible sea mango (*Cerbera odollam*) fruit through different methods of preparation either single-stage or double-stage of activation. These prepared samples were performed by physicochemical activation using phosphoric acid (H_3PO_4) and various activation atmospheres (N_2, steam, CO_2 or absence of any gases). Effect of these activation atmospheres and number of activation stages on characteristics of the resulting AC were identified by surface area, surface morphology, and surface functional group using BET analyzer, SEM, and FTIR, respectively. In this study, a few parameters have been fixed to a certain value, e.g. impregnation ratio of raw material to H_3PO_4 (1:2), heat thermal temperature (500 °C) and flow rate of the flowing gas during activation atmosphere (1.5 L/min). The efficiency of CO_2 adsorption onto the AC is evaluated *via* breakthrough time curve studies. Regeneration ability of these adsorbents modified using several amines also being studied. Therefore, this study uses four types of amine; alkanolamine (DEA and MEA), diamine (PZ) and sterically hindered amine (AMP) to be impregnated on a new AC produced from sea mango (*Cerbera odollam*) fruit with the fixed of impregnation ratio amine to AC (2:5).

1.5 Thesis Organization

This thesis is divided into 5 chapters, and a list of references.

1. **Chapter 1, Introduction**. This chapter provides the background of the study and defines the scope as well as the objectives to be achieved.
2. **Chapter 2, Literature Review**. This chapter covers the review of existing literature pertaining to the topic of the thesis.
3. **Chapter 3, Research Methodology**. This chapter is mainly a description of materials and also methods used to conduct the analysis, material preparation, data collections, and to achieve the objectives of this study.
4. **Chapter 4, Results and Discussion.** Discussion of the results obtained from the study is presented in 3 sections;

   Section 1: The characterization of the novel adsorbent.

   Section 2: The effect of impregnations on the breakthrough time of the carbon dioxide adsorption. The results explain that the different adsorption capacity found for the AC is due to the type of amine used for functionalization.

   Section 3: The regeneration analysis of these 4 amines. The regeneration ability is found to be different from one another. The structural bonding created between the reaction of CO_2 and the amino group is explained.

Section 4: The effect of flow rate and sample loadings on the breakthrough time. Different flow rates and sample loadings are tested on AMP impregnated activated carbon. The results showed that the flow rates are directly proportional to the breakthrough time.

5. **Chapter 5, Conclusion and Future Works**. In this chapter, the conclusion will be based on the results obtained will be made. The justification for the achievement of objectives is elaborated. Ideas for further study are listed in the future work section.

CHAPTER 2

LITERATURE REVIEW

2.1 Chapter Overview

This chapter provides the literature review on the properties and characteristics of activated carbon (AC) from various agricultural sources. Studies of carbon dioxide (CO_2) capture by chemically modified and non-modified AC are also reviewed.

2.2 Activated Carbon

Activated carbon is a multipurpose and frequently used adsorbents because of its tremendously high surface area and porosity, high adsorption capacity, fast adsorption kinetics, and easy regeneration (Prahas, et al., 2008). ACs are commonly used for various applications, such as purification, decolorization, filtration, separation, medical and pharmaceuticals, and wastewater and gas purification (Nasri, et al., 2014; Liu, et al., 2012).

AC has been reported to have great adsorption capacities especially in gas treatments and wastewater as a result of its well-developed porous structure and great surface area as well as variable surface chemistry (Song, et al., 2014). It is a form of carbon which encloses millions of tiny pores with interrelated capillary passages of various sizes.