AMINE–FUNCTIONALIZED KENAF AS CARBON DIOXIDE ADSORBENT IN PRESSURE SWING ADSORPTION SYSTEM

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To my beloved parents, siblings, best friends, and colleagues for their encouragements, supports, and inspirations throughout my PhD journey.

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ABSTRACT

Kenaf (*Hibiscus Cannabinus L.*) that belongs to the family of Malvaceae is abundantly grown in Malaysia since 2006 to replace tobacco plantation as it is inexpensive, easy to grow, and biodegradable. The use of kenaf as adsorbent is seen as an attractive and innovative method, and it has been used for various adsorptions. Adsorption is a promising technology that has the ability to capture carbon dioxide (CO_2), the predominant contributor of global climate change. Inspired by the established and well–known amine–based absorption process of carbon capture and storage technology, the development towards new adsorbent by introducing amine functional group has been studied. Therefore, this study explores the potential of modified kenaf as adsorbent by incorporating amine functional group on the surface and investigates the CO_2 adsorptive characteristics of amine–modified kenaf adsorbent using pressure swing adsorption system (PSA). The preparation of amine–modified kenaf was conducted via the incipient wetness impregnation technique. The physical and structural characteristics of amine–modified kenaf were determined via micromeritics 3 flex, field emission scanning electrons microscopy, energy dispersive x–ray, Fourier transform infrared spectroscopy, and thermogravimetric analyzer. The results show that the types of amine, amine loading concentration, and impregnation time affect the physical and structural properties of kenaf and thus affecting the capability for capturing CO_2. Screening of various types of amines via PSA revealed that tetraethylenepentamine (TEPA) has recorded the highest CO_2 adsorption (0.914 mmol/g). Further examination on amine loading divulged that kenaf to TEPA ratio of 1:2 presents the highest CO_2 adsorption (2.086 mmol/g) with 5 hour impregnation time. To examine the utilization of amine–modified kenaf adsorbent in PSA system, pressure bed, adsorption time, and feed flowing rate were evaluated. The result revealed that these parameters affect the gas adsorption of amine–modified kenaf adsorbent. The regeneration study had shown that kenaf adsorbent could sustain the repeated adsorption/desorption cyclic operations. This study also found that physical and chemical adsorption occurred during the adsorption of CO_2 on raw kenaf and amine–modified kenaf. Thus, amine–modified kenaf adsorbent has high potential to be used as low–cost CO_2 agro–based adsorbent hence inducing towards innovative material in the field of gas adsorption.

ABSTRAK

Kenaf (*Hibiscus Cannabinus L.*) yang berasal daripada spesies Malvacae telah ditanam secara meluas di Malaysia sejak 2006 bagi menggantikan tanaman tembakau kerana ia murah, cepat matang, dan boleh terbiodegradasi. Penggunaan kenaf sebagai penjerap dilihat sebagai kaedah yang menarik dan inovatif dan telah digunakan untuk pelbagai jenis penjerapan. Penjerapan merupakan teknologi yang menjanjikan kemampuan untuk memerangkap karbon dioksida (CO_2), iaitu penyumbang predominan kepada perubahan iklim global. Diilhamkan oleh teknologi pengumpulan dan penyimpanan karbon yang diiktiraf iaitu penyerapan berasaskan amina, pembangunan ke arah penjerap baru dengan memperkenalkan kumpulan berfungsi amina telah dikaji. Oleh itu, kajian ini mengekplorasi keupayaan pengubahsuai kenaf sebagai penjerap dengan mencantumkan kumpulan berfungsi amina pada permukaannya dan mengkaji ciri–ciri penjerapan CO_2 oleh bahan penjerap amina–kenaf menggunakan sistem penjerapan hayunan bertekanan (PSA). Penyediaan penjerap amina–kenaf dijalankan melalui teknik pengimpregnasian lembapan. Pencirian fizikal dan struktur penjerap amina–kenaf telah ditentukan melalui micromeritik 3 flex, mikroskop imbasan elektron pembebasan lapangan, sinar–x serakan tenaga, spektroskopi inframerah pengubah Fourier, dan penganalisa termogravimetrik. Keputusan kajian menunjukkan bahawa jenis amina, kepekatan amina, dan masa pengimpregnasian mempengaruhi ciri–ciri fizikal dan struktur kenaf dan keupayaan memerangkap CO_2. Saringan pelbagai jenis amina dalam PSA mendedahkan bahawa tetraethylenepentamine (TEPA) mencatat penjerapan CO_2 tertinggi (0.914 mmol/g). Kajian lanjutan terhadap kepekatan amina menunjukkan bahawa nisbah kenaf kepada TEPA 1:2 menunjukkan penjerapan CO_2 tertinggi (2.086 mmol/g) dengan masa pengimpregnasian 5 jam. Penentuan penggunaan penjerap amina–kenaf dalam sistem PSA dijalankan dengan mengkaji tekanan penjerap, masa penjerapan, dan kadar aliran masuk. Keputusan menunjukkan parameter–parameter ini telah mempengaruhi penjerapan gas oleh penjerap amina–kenaf. Kajian penjanaan semula menunjukkan bahawa penjerap kenaf dapat mengekalkan operasi kitaran penjerapan/penyahjerapan secara berulang. Kajian juga mendapati bahawa penjerapan fizikal dan kimia berlaku semasa penjerapan CO_2 pada penjerap kenaf dan amina–kenaf. Oleh itu, penjerap amina kenaf berpotensi tinggi untuk digunakan sebagai penjerap CO_2 berasaskan agro yang mempunyai kos yang rendah dan mencetus penggunaan bahan inovatif dalam bidang penjerapan gas.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AC	-	Activated Carbon
Acc. V	-	Accelerating Voltage
Al	-	Aluminium
Al_2O_3	-	Aluminium Oxide
AMP	-	2–Amino–2–Methyl–1–Propanol
Ar	-	Argon
ASEAN	-	Association of Southeast Asian Nations
Atm	-	Atmosphere
BDDT	-	Brunauer, Deming, Deming, and Teller
BET	-	Brunauer, Emmett, and Teller
BJH	-	Barrett, Joyner, and Halenda
BPL	-	Bituminous coal–based product activated carbon
BSE	-	Back Scattered Electron
BTC	-	Breakthrough Curve
C	-	Carbon
C1 / C2	-	Column 1 / Column 2
CA	-	Citric Acid
CaO	-	Calcium Oxide
CCS	-	Carbon / CO_2 Capture and Storage
Ce	-	Cerium
CFC	-	Chlorofluorocarbon
CH_3	-	Methyl group
CH_3OH	-	Methanol
CH_4	-	Methane
$(C_2H_5N)O$	-	Polyethyleneimine

C_2H_7NO	-	Monoethanolamine
$C_4H_{11}NO_2$	-	Diethanolamine
$C_4H_{11}NO$	-	2–Amino–2–Methyl–1–Propanol
$C_4H_{11}NO_2$	-	Diglycolamine
$C_4H_{13}N_3$	-	Diethylenetriamine
$C_5H_{13}NO_2$	-	Methyldiethanolamine
$C_6H_{15}N$	-	Diisopropylamine
$C_6H_{15}NO_3$	-	Triethanolamine
$C_6H_{18}N_4$	-	Triethylenetetramine
$C_8H_{23}N_5$	-	Tetraethylenepentamine
$C_{10}H_{28}N_6$	-	Pentaethylenehexamine
CMS	-	Carbon Molecular Sieve
CNT	-	Carbon Nanotubes
CO	-	Carbon Monoxide
Cu	-	Cuprum
CO_2	-	Carbon Dioxide
DE	-	Detector
DEA	-	Diethanolamine
DETA	-	Diethylenetriamine
DGA	-	Diglycolamine
DIPA	-	Diisopropylamine
E–100	-	Ethyleneamine
EDX	-	Energy Dispersive X–ray spectroscopy
EPU	-	Economic Planning Unit
ETP	-	Energy Technology Perspectives
Fe	-	Iron
FESEM	-	Field Emission Scanning Electrons Microscopy
FID	-	Flame Ionization Detector
FTIR	-	Fourier Transform Infrared spectroscopy
GC	-	Gas Chromatography
GDP	-	Gross Domestic Product
GFC	-	Gas Flow Controller
GHG	-	Greenhouse gas
H^+	-	Hydrogen Ion

H_{ads}	-	Heat of adsorption
H_2	-	Hydrogen
H_2O	-	Water or moisture content
HCl	-	Hydrochloric Acid
HCO_3^-	-	Bicarbonate Ion
He	-	Helium
IEA	-	International Energy Agency
IPCC	-	Intergovernmental Panel on Climate Changes
IR	-	Infrared
IUPAC	-	International Union of Pure and Applied Chemistry
K	-	Potassium
K_2HPO_4	-	Dipotassium Phosphate
KBr	-	Potassium Bromide
KOH	-	Potassium Hydroxide
MAXSORB	-	High Surface Area Active Carbon
MB	-	Methylene Blue
MCM	-	Mobile Crystalline Material
MDEA	-	Methyldiethanolamine
MEA	-	Monoethanolamine
MMEA	-	Monomethylethanolamine
Mn	-	Manganese
MOF	-	Metal–Organic Framework
MS	-	Mass Spectrometry
MTZ	-	Mass Transfer Zone
Mw	-	Molecular Weight
N_2	-	Nitrogen
N_2O	-	Nitrous Oxide
NFESC	-	Naval Facilities Engineering Service Center
NH_2		Amine functional group
NH_3	-	Ammonia
NKTB	-	National Kenaf, and Tobacco Board
NO_2	-	Nitrogen Dioxide
NRE	-	Ministry of Natural Resources and Environment
O_2	-	Oxygen

OH	-	Hydroxyl group
PAC	-	Palm Activated Char
PEHA	-	Pentaethylenehexamine
PEI	-	Polyethyleneimine
pH	-	Power of Hydrogen
PKC	-	Palm Kernel Char
PMMA	-	Polymethylmethacrylate
Ps	-	Polystyrene
PSA	-	Pressure Swing Adsorption
R / R’	-	Alkyl group
$RR'NCOO^-$	-	Carbamate Ion
$RR'NH_2^+$	-	Ammonium Ion
R & D	-	Research and Development
Ret. Time	-	Retention Time
SBA	-	Santa Barbara Amorphous
SCFH	-	Standard Cubic Feet per Hour
SE	-	Secondary Electron
SEM	-	Scanning Electron Microscopy
Si	-	Silanol
SiO_2	-	Silicon Dioxide
SMR	-	Steam Methane Reformer
SO_2	-	Sulphur Dioxide
TCD	-	Thermal Conductivity Detector
TEA	-	Triethanolamine
TEPA	-	Tetraethylenepentamine
TEPAN	-	Tetyraethylenepentaamineacrylonitrile
TETA	-	Triethylenetetramine
TGA	-	Thermogravimetric Analysis
TPD	-	Temperature Programmed Desorption
TPR	-	Temperature Programmed Reduction
TSA	-	Temperature Swing Adsorption
V_1	-	Valve 1
VSA	-	Vacuum Swing Adsorption
WD	-	Working Distance

WEO	-	World Economic Outlook
XPS	-	X–ray Photoelectron spectroscopy
XRD	-	X–ray Diffraction spectroscopy
$ZnCl_2$	-	Zinc Chloride

LIST OF SYMBOLS

α	-	Accommodation coefficient and selectivity
A	-	Area of column
Å	-	Angstrom
D	-	Column diameter
k	-	Henry's Law constant
K'	-	Henry's Law constant
H	-	Isosteric heat of adsorption
i / j	-	Types of components
m	-	Mass of adsorbent
P	-	Total pressure
P_o	-	Initial pressure
P_{ads}	-	Pressure adsorption
P_{des}	-	Pressure desorption
p	-	Partial pressure
ΔP	-	Pressure drop
ρ	-	Gas density
Q / q	-	Gas adsorption capacity
R	-	Universal gas constant (8.314 J/mol.K)
T	-	Absolute temperature
T_{ads}	-	Temperature adsorption
T_{bp}	-	Temperature boiling point
T_{des}	-	Temperature desorption
T_{max}	-	Main degradation step
τ	-	Time
t_b	-	Breakthrough time

t_e	-	Exhaustion time
U	-	Internal Energy
v	-	Gas velocity
V	-	Volumetric rate
x	-	Degradation temperature
y	-	Weight residue (%)
y_o	-	Mole fraction
θ	-	Surface coverage
Δ	-	Deviation

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Adsorption processes have been utilized since 1950s in removing a wide variety of organic vapor and inorganic gas. The applications of adsorption processes have been expanding due to the inventions in designing the system and development of improved adsorbent. Gas adsorption phenomena could be defined as a result of the intermolecular force interaction that exists between the gas molecules on the exposed surface of solid material (Dabrowski, 2001). The gas molecules and the surface solid material are indicated as the adsorbates and adsorbent, respectively. The attraction of adsorbates on the surface of adsorbent could be achieved via physical and chemical adsorption mechanisms. A physical adsorption (physisorption) involves the relatively weak intermolecular force of adsorbates–adsorbent such as dispersion, dipolar or van der Waals interactions. Conversely, chemical adsorption (chemisorption) is achieved through the strong interaction of carbon dioxide (CO_2) molecules on the surfaces of adsorbent by the substantial sharing of electrons to create covalent or ionic bonding. As a consequence of adsorbates–adsorbent interaction, the gas molecules near to the solid adsorbent experience reduction in the potential energy and lead to concentrate; so that the molecular density becomes greater than in the free–gas phases (Ruthven *et al.*, 1994).

The past two decades has seen tremendous growth of adsorption systems particularly for gas separation and purification process (Barrer, 1978; Suzuki, 1990; Yang, 1997). The adsorption applications for gas separation and purification includes the separation and purification of hydrocarbons from the vent stream, hydrogen from steam reformers, carbon dioxide from natural gas stream, removal of pollutants from flue gases, and purification of mercury from cell gas effluent (Keller, 1983). Besides, separation process is generally accounted as a major production cost in the chemical and petrochemical industries. According to King (1980), separation process based on the adsorption is affected by a mass separating agent, which is adsorbent. Therefore, the performances of adsorption process could be directly governed by the quality of the adsorbent used. A handful of generic adsorbents that dominates the commercial applications of adsorption fields are activated carbon, zeolite, silica gel, and activated alumina (Yang, 2003). Ideally, the adsorbent used must be tailored with the specific attributes to suit with the specific applications. However, the development of better adsorbent that has high potential for the adsorption and improves the performance of the current commercial process is scarce. Therefore, the invention towards a potential adsorbent remained as the attractive task amongst researchers worldwide.

The exploration towards agro–based adsorbent to replace the commercialized existing adsorbents has been explored as it is widely available, renewable, potentially less harmful, benign to the environment and cost–effective. Biomass used is typically obtained from waste material or by–product from commercial activities since the amount of waste yielded from agriculture sector has progressively increased due to rapid industrial development. Recently, the utilization of agricultural source is seen as an inventive approach and considered as a promising adsorbent for gas adsorption application (Othman and Akil, 2008). Previous studies have reported that coffee ground, almond shell, olive stone, african palm shell, coconut shell, bamboo, and kenaf has a vast potential for gas adsorption application (Plaza *et al.*, 2012; González *et al.*, 2013; Nasri *et al.*, 2013; Ello *et al.*, 2013; Vargas *et al.*, 2012; Othman and Akil, 2008). However, the application of these agriculture products is limited for the production of activated carbon fiber. Thus, the current innovation and development on the agriculture sources remain as challenging tasks to be executed.

1.2 Current Problems and Future Prospects

Recently, the rising concentrations of carbon dioxide (CO_2) in the atmosphere are linked with the global climate changes (Yang *et al.*, 2008). The fourth assessment report of the Intergovernmental Panel on Climate Changes (IPCC's) has publicized that the global atmospheric concentration, resulted from anthropogenic CO_2 emission has increased from a preindustrial level (~280 ppmv) to 379 ppmv in 2005 and keeps increasing up to ~390 ppmv (Pachauri and Reisinger, 2007; Oh, 2010). Based on this report, the efforts to reduce the concentration of CO_2 emitted to the atmosphere have been implemented actively and vigorously. The sequestrations of CO_2 under the CO_2 Capture and Storage (CCS) technology is identified as a major option to address the global warming and climate change issues. The CCS technology has been established in Malaysia since the Intergovernmental Panel on Climate Change (IPCC) in 2007. It has been predicted that the average global surface temperature will rise up to 2^{o}C in between 1990 and 2100. Thus, CCS project is seen as a key technology that would assist Malaysia in achieving the goal to combat with the global demands in reducing the carbon emission by 40 % in 2020 based on 2005 level (Yu *et al.*, 2012). The primary steps of CCS technologies are CO_2 capture, compression, transportation, and storage system. The CO_2 capturing is executed at a fixed point source such as power plants and cement manufacturing facilities with the appropriate strategies. In order to achieve an economical sequester, it is a decisive alternative to have the cost–effective capture technology in relatively highly concentrated stream prior to compressed CO_2 at very high pressure.

The CCS technology can be achieved via a variety of techniques including amine–based liquid absorption, solid adsorption by adsorbents, cryogenic distillation techniques, and selective diffusion through polymer, ceramic or metallic membranes (Gray *et al.*, 2005; Zanganeh *et al.*, 2009; Brunetti *et al.*, 2010). Amongst these techniques, amine–based chemical absorption process using amine aqueous solution is the most favoured method and has been widely practiced. However, this existing technology suffers from several disadvantages such as high energy intensive for regeneration process, the needs for large absorber volume, low contact area between gas and liquid thus results to the low CO_2 loading, thermal and oxidative degradation of solvent, and produce corrosive product (Resnik, 2004; Haszeldine, 2009; Samanta *et al.*, 2011). Subsequently, this phenomenon induced the researchers and scientists worldwide to explore the cost–effective technologies for capturing CO_2. Therefore, solid adsorption has been proposed as a promising alternative separation technology as it promotes numerous potential advantages such as high adsorption capacity, low energy for regeneration, low equipment cost, avoid corrosion problem, and ease of applicability over a relatively wide range of temperature and pressure (Duffy *et al.*, 2006; Mandal and Bandyopadhyay, 2006; Plaza *et al.*, 2007; Serna–Guerrero *et al.*, 2010; Samanta *et al.*, 2011).

The success of this approach relies on the development of adsorbent material that meet crucial characteristics such as high CO_2 adsorption capacity and selectivity, demonstrates microstructure and morphological stability, infinite regenerability and stability and relatively fast adsorption/desorption kinetics under operating condition, and cost–effective (Yong *et al.*, 2002; Zheng *et al.*, 2007; Choi *et al.*, 2009; Sayari *et al.*, 2011). A variety of solid adsorbents that have been reported by previous studies show high affinities towards CO_2 including porous carbonaceous materials, activated carbon (ACs), zeolites, alumina silica, silica gels, metal–organic framework (MOF), carbon nanotubes (CNT), carbon molecular sieves (CMS), lithium zirconate, hydrotalcite, calcium oxide (CaO), and organic–inorganic hybrids (Choi *et al.*, 2009; Samanta *et al.*, 2011). However, exploring toward better and improved adsorbent that is developed according to the fundamental principles and not commercially available is quite challenging. Therefore, selection of green material to replace the utilization of the existing commercial solid adsorbents is seen as a future prospect that should be explored by researchers and scientists since natural material is potentially less harmful, benign to the environment, has high affinity to the organic molecule mainly solvent and chemical, operates well under wide range of temperature and humidity level, inert, safe to handle, highly available, and cost–effective material (Othman and Akil, 2008).

1.3 Problem Statement

The adsorption force is associated to the natural phenomenon that happens between adsorbed molecules (adsorbates) and adsorbent. The different adsorbates are attracted to the adsorbent with different affinities which is known as “selectivity” (Ruthven *et al.*, 1994). Thus, a selection of an appropriate adsorbent is a crucial stage that should be considered in the adsorption study. The selected adsorbent should possess high selectivity and high adsorption capacity towards the adsorbates, good mechanical strength, has adequate adsorption and desorption kinetic energy, has thermal, chemical and mechanical stabilities under repeated cyclic operations, low heat capacity, high porosity, and cost–effective material (Yang, 1997; Yong *et al.*, 2002; Yu *et al.,* 2012). Therefore, various commercial and established adsorbents have been explored by previous researchers for capturing CO_2. However, some of the adsorbents have low capacity of CO_2 adsorption due to high hydrophilic character in a presence of moisture, requires high regeneration temperature (Wang *et al.*, 2011; Sayari *et al.*, 2011), has low CO_2 adsorption capacity at atmospheric pressure (Liu *et al.*, 2005; Sun *et al.*, 2007; Chew *et al.*, 2010), the adsorption capacity reduced when expose to a mixture of gases, and exhibits exceptional CO_2 adsorption capacity with CO_2 at high pressures (Millward and Yaghi, 2005). Moreover, the estimated cost for these adsorbents is too high for the large–scale applications.

To overcome some of these drawbacks, exploration and investigation towards the development of potential adsorbents based on natural material that is less harmful and more benign to environment becomes an alternative option amongst researchers worldwide. Hereinafter, the increasing attention on the utilization of natural materials in the adsorption engineering has inspired the study to reveal the potential of natural material for the CO_2 adsorption. Additionally, the motivation and incentives given by government to intensify the use of kenaf in Malaysia is in line with the demands for the natural adsorbent originated from agro–based material. Previous studies on kenaf have acknowledged that kenaf has high potential as a natural adsorbent for different types of adsorbates such as carbon dioxide (Othman and Akil, 2008), water (Zaveri, 2004; Lips *et al.*, 2009), methylene blue (Mahmoud *et al.*, 2011), oil (Othman *et al.*, 2008), and heavy metal ions (Sciban *et al.*, 2007; Othman *et al.*, 2008; Aber *et al.*, 2009; Garcia *et al.*, 2012). However, all these reported journals were explored on the utilization of carbonaceous kenaf and/or activated carbon kenaf. It is quite interesting to investigate the potential of raw kenaf without undergoing carbonization step but only implementing chemical impregnation on raw kenaf for the CO_2 adsorption since no literatures are stated based on this invention. In fact, the modification of chemical based kenaf adsorbent is costly and energy effective; thus a research on its potential towards CO_2 adsorbate is most welcomed.

The modification of porous material adsorbent by selecting functional groups via impregnation or grafting method have been recommended in order to improve the CO_2 adsorption capacity and to promote the mass transfer rate of CO_2 into the porous adsorbents (Yu *et al.*, 2012). According to Samanta *et al.* (2011), the modification of adsorbent surface can be achieved by using two common functional groups; alkaline carbonate and amine. Inspired by the most applicable technologies of amine–based chemical absorption for capturing CO_2, the development of potential adsorbent based on amine–functionalize group is an alternative method to overcome the problems due to this conventional technique such as low contact area between gas and liquid, low CO_2 loading, and severe absorbent corrosions (Yu *et al.*, 2012). The incorporation of amine functionalize group on the adsorbent surface is believed can improve the basic active sites which facilitates the interaction towards acidic CO_2 adsorbate. Moreover, the incorporation of amine functional group on adsorbent support either via ammonia heat treatment or chemical impregnation technique offers high adsorption capacities, reduce the consumption of energy for regeneration step, and avoid corrosion problem as well as evaporation of amines (Maroto–Valer *et al.*, 2005; Aziz *et al.*, 2012; Khalil *et al.*, 2012). Nevertheless, its' usage is only limited to the micropore and mesopore adsorbents. For this reason, research interest for the production of amine–modified adsorbent from agro–based materials grows rapidly in recent and remain in challenge for the researchers' and scientists' worldwide.

1.4 Research Objectives

Based on the outlined problem, research in the field of gas adsorption study is diverse with the applications of kenaf as a potential CO_2 adsorbent. The objectives of this study are:

1. To modify the physical and structural properties of kenaf
2. To determine the physical and structural properties of kenaf adsorbent
3. To measure the CO_2 adsorption and regeneration of kenaf adsorbent

1.5 Research Scopes

To be an applicable and competitive CO_2 adsorbent, the adsorption properties of kenaf should be improved. The CO_2 adsorption and selectivity could be improved via two alternative strategies, modifying the physical and structural properties of the adsorbent. The surface properties of the sorbent was varied by introducing different types of amines namely monoethanolamine (MEA), diethanolamine (DEA), methyldiethanolamine (MDEA), 2–amino–2–methyl–1–propanol (AMP), polyethyleneimine (PEI), diethylenetriamine (DETA), triethylenetetramine (TETA), tetraethylenepentamine (TEPA), diisopropylamine (DIPA), pentaethylenehexamine (PEHA), triethanolamine (TEA), and diglycolamine (DGA). The modification technique was achieved via wetness impregnation method. This screening procedure was carried out to determine the suitable modifier for capturing CO_2 adsorbates. The amine loading and impregnation time would also be varied to determine the suitable conditions for amine–impregnation.

The understanding of the physicochemical and structural characteristics of the adsorbent is very important to improve the fundamental knowledge of the adsorption process. Extensive investigation based on the fundamental aspect were included to attain a better understanding on the structural and gas adsorptive properties of kenaf adsorbent for gas adsorption study. Based on the second objective, the changes in the surface morphology, elemental content, chemical bonding, and degradation weight of kenaf were analyzed via Field Emission Scanning Electrons Microscopy (FESEM), Energy Dispersive X–ray (EDX), Fourier Transform Infrared spectroscopy (FTIR), and Thermogravimetric Analyzer (TGA), respectively. The analysis on the physical properties such as surface area, pore volume, and pore size were conducted based on the nitrogen adsorptive analysis at –196^{o}C (77 K). The gas adsorptive properties of kenaf were studied to understand the adsorbates–adsorbent interaction mechanisms.

To complete the research objective, the CO_2 adsorption and regeneration study was accomplished in the pressure swing adsorption (PSA) system. PSA system is selected because it is a versatile, energy–efficient, and cost–effective technology for the CO_2 separation and purification process over the conventional gas separation technologies. In this study, the adsorption process was conducted by using four steps of dual–columns of PSA system at pressure of 1.5 bar and varied up to 4 bar. The adsorption study in PSA system involves several parameters such as adsorption time, pressure bed, and feed flowing rate. Besides, the screening of amine–modified kenaf was also carried out in single column by varying the effects of particle sizes, types of amines, amine loadings, and impregnation time. Hereinafter, the regeneration process has achieved up to ten (10) cycles of operations for 60 minutes (1 hour). This study used the purified CO_2 and N_2 (99.999 %), and the gas mixture of CO_2/N_2 in a ratio of 30/70.

1.6 Significant of Study

The finding of this study explored the potential of amine-modified kenaf for the CO_2 adsorption and separation. Thus, this study will be a significant endeavor in promoting a new strategy to diversify the utilization of kenaf as an adsorbent. It is noticed that the global climate changes resulted from the emissions of anthropogenic greenhouse gas, and CO_2 become the inspiration and motivation towards the validity of this research since CO_2 is increased significantly from the pre–industrial level up to recent years. As a result, the idea to improve the ***applications of natural source as adsorbent in gas adsorption field*** is seen as an attractive, innovative, and significant task to be conducted. The introduction of kenaf as a potential green–based adsorbent for the gas adsorption has intensified the broad application of kenaf since Malaysian government has allocated about RM 65 million under 10th Malaysia Plan for research and development (R & D) on kenaf developments. From the practical standpoint, this study is very relevant and pertinent to be implemented in Malaysia as kenaf is easily accessible particularly in Kedah and Kelantan. Consequently, the innovation towards material for gas adsorbent is one way forward in this study.

To improve the adsorptive and regenerative behaviors of kenaf adsorbent, the additional treatments have exerted on its surface. For the reason, kenaf adsorbent has been treated using basic organic group of amine–based chemical. Thus, the particular interests on the ***modifications of kenaf adsorbent using amine–based functionalized groups*** were identified as major contribution of this research since amine aqueous solution has been widely applied for capturing CO_2 in the industrial and commercial purpose up to these recent days. The incorporation of amine functionalized groups on kenaf surface structure provides the basic active sites that are beneficial to attract the CO_2 adsorbates hence facilitate the adsorption capacity of kenaf adsorbent. Besides, the invention of amine–based functionalized group on kenaf surfaces is an alternative over a well–known physical modification of adsorbent, activated carbon that requires high temperature for activation. Then, the CO_2 adsorption study for amine–modified kenaf was performed in a versatile system namely pressure swing adsorption (PSA).

1.7 Research Limitations

There are some unavoidable limitations for this study. Firstly, the physical and structural characterizations study of kenaf sample is limited for the FESEM, EDX, FTIR, TGA, and nitrogen analysis at 77 K (–196^{o}C). There is a lot of powerful characterization analysis which are beneficial to elucidate the chemical dispersion as well as the reducibility of deposited amine of the samples. However, availability of equipment limits the characterization analysis. This research only involves limited studies of CO_2 adsorption in PSA system; and the thermodynamic and kinetic studies are not included. Finally, the adsorption parameter in PSA system is limited for the variations of pressure bed, adsorption time, and feed flowing rate.

1.8 Thesis Outlines

The contents of thesis consist of five chapters that are presented in sequential order. Initially, **Chapter 1** provides brief introduction on the adsorption process and the applications. A variety of adsorbents dominating the commercial applications of adsorption field work was mentioned as a research background. The invention of green–based adsorbent originated from natural source is a dominant contribution of this study. To meet new challenging research task, this chapter highlights three main objectives; where the introduction of green–based material for adsorption separation process plays a significant and relevant role. The objectives of study were developed based on the aforementioned current problems and research backgrounds which are also included in Chapter 1.

Chapter 2 highlights the literature review of several aspects associated to the fields of research studies. The issue of global climate change contributed by carbon dioxide (CO_2) becomes the main focus. Review on the introduction of kenaf as green–based adsorbent is essential to be considered. Besides, Chapter 2 also reviews on the specification of adsorbent, existing commercial adsorbent, potential adsorbent for capturing CO_2, fundamental theory of gas adsorption process, and pressure swing adsorption (PSA) engineering system. A literature review of PSA involves the basic principle of the PSA, elementary step of the cycle, and application of PSA system for various adsorbents.

Furthermore, **Chapter 3** discusses on the material and methodology used throughout the experimental study. Chapter 3 lists the general material, chemicals, and gases used in the experimental procedures to accomplish the research objectives. The analytical technique used to determine the structural and physical characteristics of the raw kenaf and amine–modified kenaf adsorbent are also described in Chapter 3. Moreover, the base operating conditions in PSA system and procedures to conduct the adsorption and regeneration steps are also clarified in this chapter. The operation of gas chromatography (GC) is also included in Chapter 3.

The results are shown and discussed in **Chapter 4**. In general, the discussions are divided into two parts. The first part explains the determination of structural and physical characterizations analysis of kenaf and amine–modified kenaf adsorbents by using several analytical techniques. The second part discusses on CO_2 adsorption and regeneration process in a pressure swing adsorption (PSA). At the end of Chapter 4, the potential of raw kenaf and amine–modified adsorbent for the CO_2 adsorption is compared with several reported agro–based adsorbents. Finally, **Chapter 5** presents the conclusions and recommendations for future work.

1.9 Summary

The rising CO_2 concentration in the atmosphere is linked with global climates change. According to this fact, the sequestration of CO_2 under CCS technologies is acknowledged as the main preference to address the critical environmental issues. A recent study reveals the contribution of amine–modified kenaf adsorbent for the CO_2 adsorption applications. A study on the adsorption properties of kenaf explains about its potential in terms of adsorption, regeneration, and selectivity characteristics. This study was succeeded by explicating the interaction of CO_2 adsorbate on kenaf; so the adsorbates–adsorbent interactions concept is well understood. Finally, the invention and formulation of agro–based adsorbent that offers high potential for gas adsorption is in demand for the subsequent era. Therefore, the active research efforts should be established in the future to commercialize this new invention.

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