MODIFICATION OF COCONUT SHELLS BY POLYETHER ETHER KETONE FOR HIGH-PRESSURE ADSORPTION OF METHANE AND NATURAL GAS

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To the almighty Allah, the most Beneficent, the most Merciful. Also to my parent and all my extended family members.

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ABSTRACT

Application of natural gas (NG) as a transportation fuel introduces the possibility of reducing the dependency of liquid based petroleum fuel and emissions of greenhouse gases. At present, compression and liquefaction are the most used technology used for transportation system. For transportation use, compression requires high pressure (200–300 bar) while liquefaction is impractical. A relatively low pressure of 30-40 bar is achievable by adsorbed natural gas to store nearly compressed natural gas. In this study, adsorbents for high-pressure adsorption of methane (CH_4) and NG were prepared from coconut shells and polyether ether ketone (PEEK) using potassium hydroxide (KOH) and microwave-assisted activation. The role of KOH was to modify the properties and structure of the adsorbent to suffice better adsorption applications. Design expert software version 7.1.6 was used for optimization and prediction of preparation conditions of the adsorbents for CH_4 and NG adsorption. Effects of microwave power, activation time and quantity of PEEK on the adsorbents performance toward CH_4 and NG adsorption were investigated. The adsorbents were characterized by pH, Fourier transform infrared spectroscopy, thermogravimetric and derivative thermogravimetric, mechanical property, nitrogen adsorption, bulk density, scanning electron microscopy, and ultimate and proximate analyses. The ideal CH_4 and NG adsorption capacities of adsorbents were determined using volumetric method at pressures of 5, 7.5, 11, 17, 25, 30 and 35 bar at ambient temperature, while at 5 ^oC, the adsorption was carried out at 5, 17 and 35 bar. Isotherm and kinetics models were used to validate the experimental results. The optimum preparation conditions were found to be 15 wt % of PEEK, 3 minutes activation time and 300 W microwave power. The highest CH_4 uptake of 9.7045 mmol CH_4 adsorbed/g adsorbent was recorded by adsorbent (M33P15) (300 W of microwave power, 3 minutes activation time and 15 wt % amount of PEEK) among the sorbents at ambient temperature and 35 bar. Similarly, the highest NG uptake of 9.9432 mmol NG adsorbed/g adsorbent was also achieved by the same sample under the same adsorption conditions. The CH_4 and NG equilibrium data were well correlated with Sips, Toth, Freundlich and Langmuir. Isotherms revealed that the Sips isotherm has the best fit, while the kinetics studies revealed that the pseudo-second-order kinetic model best describes the adsorption process. In all scenarios studied, a decrease in temperature led to an increase in adsorption of both gases. The M33P15 maintained its stability even after seven adsorption/desorption cycles. The findings revealed the potential of coconut shell-PEEK as CH_4 and NG adsorbent.

ABSTRAK

Pengaplikasian gas asli (NG) sebagai bahan api pengangkutan memberi kemungkinan pengurangan terhadap kebergantungan kepada bahan api berasaskan petroleum cecair dan pengurangan terhadap pelepasan gas rumah hijau. Pemampatan dan pencairan adalah teknologi yang paling banyak digunakan untuk sistem pengangkutan sehingga kini. Secara relatifnya tekanan yang tinggi (200–300 bar) diperlukan bagi pemampatan manakala pencairan tidak dipraktikkan untuk kegunaan pengangkutan. Tekanan rendah dari 30-40 bar boleh dicapai oleh gas asli terserap untuk menyimpan gas asli termampat. Dalam kajian ini, penjerap-penjerap untuk penjerapan metana (CH_4) bertekanan tinggi dan NG telah disediakan daripada tempurung kelapa dan polieter-eter-keton (PEEK) dengan menggunakan kalium hidroksida (KOH) dan pengaktifan berbantukan gelombang mikro. Peranan KOH adalah untuk mengubahsuai sifat-sifat dan struktur bahan penjerap untuk meningkatkan aplikasi kadar penjerapan yang lebih baik. Perisian design expert versi 7.1.6 telah digunakan untuk pengoptimuman dan ramalan keadaan penyediaan penjerap-penjerap untuk penjerapan CH_4 dan NG. Kesan-kesan daripada kuasa gelombang mikro, masa pengaktifan dan kuantiti PEEK ke atas prestasi penjerap-penjerap terhadap penjerapan CH_4 dan NG telah dikaji. Penjerap-penjerap dicirikan menggunakan pH, inframerah transformasi Fourier, analisis termo-gravimetri dan termo-gravimetri derivatif, sifat mekanikal, penjerapan nitrogen, ketumpatan pukal, mikroskop imbasan elektron dan analisis proksimat dan analisis muktamad. Kapasiti penjerapan penjerap-penjerap CH_4 dan NG yang ideal telah ditentukan menggunakan kaedah isipadu pada tekanan 5, 7.5, 11, 17, 25, 30 dan 35 bar pada suhu ambien sementara pada 5 ^{o}C, penjerapan dijalankan pada 5, 17 and 35 bar. Model kinetik dan isoterma telah digunakan untuk mengesahkan hasil eksperimen. Keadaan penyediaan optimum telah didapati iaitu 15 wt % jumlah PEEK, 3 minit masa pengaktifan dan 300 W kuasa gelombang mikro. Penjerapan penjerap CH_4 yang paling tinggi iaitu 9.7045 mmol CH_4 terjerap/g telah direkodkan oleh penjerap (M33P15) (300 W kuasa gelombang mikro, 3 minit masa pengaktifan dan 15 wt % jumlah PEEK) di antara penjerap-penjerap yang lain pada suhu ambien dan 35 bar. Begitu juga, penjerapan penjerap NG yang paling tinggi iaitu 9.9432 mmol NG terjerap/g juga telah dicapai oleh sampel yang sama dalam keadaan penjerapan yang sama. Data keseimbangan CH_4 dan NG adalah berkorelasi baik dengan Sips, Toth, Freundlich dan Langmuir. Isoterma-isoterma mendedahkan bahawa isoterma Sips adalah yang paling padan manakala kajian kinetik mendedahkan bahawa model kinetik pseudo-tertib-kedua adalah yang terbaik untuk menerangkan proses penjerapan. Dalam semua keadaan kajian, pengurangan pada suhu membawa kepada peningkatan pada penjerapan kedua-dua gas. M33P15 mengekalkan kestabilan walaupun selepas tujuh kitaran penjerapan/penyahjerapan. Penemuan-penemuan mendedahkan potensi tempurung kelapa-PEEK sebagai penjerap CH_4 dan NG.

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a	-	Adsorption cell
AC	-	Activated carbon
ANG	-	Adsorbed natural gas
ANOVA	-	Analysis of variance
BET	-	Branauer Emmett Teller
CNSC	-	Coconut shells char
CPBC	-	Coconut-PEEK blended char
CCD	-	Central composite design
CCS	-	Carbon capture and storage
CDM	-	Clean development mechanism
CER	-	Certified emission reduction
CFCs	-	Chlorofluorocarbons
CNG	-	Compressed natural gas
CNTs	-	Carbon nanotubes
D_{avg}	-	Average pore size
DTG	-	Derivative thermo-gravimetric
3-D	-	3 Dimensional
EOR	-	Enhanced Oil Recovery
ERUs	-	Emission reduction units
FC	-	Fixed carbon
FTIR	-	Fourier transform infrared spectroscopy
GHG	-	Greenhouse gas
JI	-	Joint implementation
K_{int}	-	Intra-particle diffusion rate constant

K_F	-	Freundlich constant
K_L	-	Langmuir constant
K_{LF}	-	Sips constants
KBr	-	Potassium bromide
l	-	Loading cell
LNG	-	Liquefied natural gas
M	-	Molar mass
m	-	mass
MOFs	-	Metal organic frameworks
MWCNTs	-	Multiwall carbon nanotubes
n	-	Adsorption intensity
N	-	Number of experiment
NGV	-	Natural gas vehicle
P	-	Pressure
PC	-	Porous carbon
PCs	-	Porous Carbons
PEEK	-	Polyether Ether Ketone
PEKC	-	Polyether Ether Ketone char
q	-	Amount adsorbed
q_m	-	Maximum amount adsorbed
q_d	-	Desorption capacity
q_e	-	Adsorption equilibrium
q_{exp}	-	Experimental amount adsorbed
q_p	-	Predicted amount adsorbed
q_r	-	Desorption rate
q_t	-	Amount adsorbed at time t
R^2	-	Correlation coefficient
R^2 adj	-	Correlation coefficient adjusted
RSM	-	Response surface methodology
RSMD	-	Root mean square deviation
R&D	-	Research and Development
SEM	-	Scanning electron microscopy
S_{BET}	-	Surface area, BET

TG	-	Thermo-gravimetric
V	-	Volume
VM	-	Volatile matter
VOCs	-	Volatile organic hydrocarbons
V_{micro}	-	Micropore volume
V_{tot}	-	Total pore volume
W	-	Watts
W_c	-	Weight of char
W_p	-	Weight of precursor
W_{pc}	-	Weight of porous carbon
XRD	-	X-Ray diffraction
Y_{pc}	-	Yield of porous carbon
Y_c	-	Yield char

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

With the recent concern about the emissions from diesel engines, in addition to the increasing demand for energy coupled with instability of conventional fuel prices, has influenced interest towards sourcing alternative fuel (Wei and Geng, 2016). Many countries want to reduce their dependency on the imported fuels (diesel and gasoline) due to the aforementioned issues. These issues could be solve by initiating a reliable and visible technology that can accommodate the demands in the energy application sectors. Works by Wang *et al.* (2017) stated that the available and reliable energy that can secure sustainability in environmental delivery with minimal Green House Gases (GHGs) emission is Natural Gas (NG). With increasing government interest in restriction of GHGs emission from transportation systems and growing concern over the creation of sustainable environment by adaption of alternative fuel known as NG has gained popularity (Yang *et al.*, 2014a).

Greenhouse Gases (GHGs) emission has attracted global attention due to their negative contribution to the environment. In order to control its negative contribution to the biosphere, the international community has initiated an incentive, policies, regulations, and agreements that governed the GHG's emission under Kyoto protocol. These include Joint Implementation (JI), emission-trading known as carbon market and Clean Development Mechanism (CDM). United Nation Framework Convention on Climate Change (UNFCCC), 2013, stated that the parties were fully engaged and committed to lowering the emission of GHGs in the years to come (2013 to 2020) by 18 percent below the year 1990 emission level. The commitment is paying attention to six GHGs, which include; nitrous oxide (N_2O), sulfur hexafluoride (SF_6), perfluorocarbons (PFCs), hydrofluorocarbons (HFCs), methane (CH_4) and carbon dioxide (CO_2) (Guo and Liang, 2016).

The two pronounced methods for NG storage are in liquefaction state and in compression form (El-Sharkawy *et al.*, 2015). The major setback of liquefied natural gas (LNG) stored at low temperature (112 K) which requires special equipment (He *et al.*, 2013). Compressed natural gas (CNG) stored at 200 to 300 bar (El-Sharkawy *et al.*, 2015) in which expensive/heavy operating equipment are used, limited refueling stations and the high cost of refueling with no safety guarantee to passengers on board (Beckner and Dailly, 2015). Adsorbed natural gas (ANG) storage system is been considered as a promising and the best option.

Previously, adsorbent such as zeolites, silica gels, metal organic frame work and mesoporous solids for methane storage has been reviewed (Menon and Komarneni, 1998). Some of these adsorbents exhibit high-adsorption performance in CH_4 but they are expensive and require multi-stage fabrication. Carbon materials are more suitable for methane adsorption due to their easy tailoring texture and surface properties, low cost, availability, low energy requirement and hydrophobicity (Labus *et al.*, 2014).

Coconut shell is the leading raw material used for the production of activated carbon in Asia, which is over 60% share of the total raw materials used (Bandosz, 2006). This might be due to its availability around the region and possession of excellent properties such as faster kinetic, high carbon content, low organic matter, hardness and high storage capacity (Sahoo and Ramgopal, 2016). In addition, it can withstand higher-pressure operations with several cycles of reusability (Sahoo and Ramgopal, 2016). A lot of by-products are usually generated in the form of fibers or shells annually. Therefore conversion of the by-product to activated carbon is imperative due to the current environmental problem.

Coconut shells activated carbon indeed, showed a good CH_4 and natural gas adsorption performance in numerous conditions. However, it also suffers from instability issues, due to the poor mechanical properties at higher pressure, which can lead to the inability of the activated carbon to withstand the high-pressure adsorption. This issue is typically overcome by blending the coconut shells with structural polymer, namely Polyether Ether Ketone (PEEK), which adds more strength to the activated carbon matrix under high-pressure condition, while facilitating the gas transport mechanism. The blending purpose is to obtain a positive result in terms of stability and adsorption performance.

The conventional heating method for production of activated carbon is by using a furnace. The technique generates high temperature which will lead to consumption of large amount of energy, making it less effective and very expensive (Hesas *et al.*, 2013b). An alternative technique is microwave activation achieved at a lesser heating period compared to the conventional type of activation. Microwave is an energy efficient and cost effective equipment for producing activated carbons with higher surface area and better yield compared to conventional heating equipment (Hamza *et al.*, 2015; Hesas *et al.*, 2013b). The proposed microwave-assisted for the activation of activated carbon in this work because of the above-highlighted advantages.

Several chemicals have been used as an activating agents in the production of activated carbon, which includes; phosphoric acid (Hesas *et al.*, 2013b), sodium hydroxide (Auta *et al.*, 2013), zinc chloride (Ozdemir *et al.*, 2014), potassium carbonate (Jin and Zhu, 2014), potassium hydroxide (Nasri *et al.*, 2014; Tseng and Tseng, 2005) and so on. Among these activating agents, potassium hydroxide proved to be the best chemical for activation due to its ability to develop adsorbent with higher surface area and larger pore size, influenced from the intercalation of potassium metal with carbon material (Auta and Hameed, 2011). The synthesis conditions and routes also play an important role (Gadipelli and Guo, 2015). This, therefore, call the attention of researchers towards exploring more efficient and effective processing routes for activated carbon production.

This research work focus towards the production of novel activated carbon from coconut shells as one of the most available biomass in Malaysia with PEEK as a support material and potassium hydroxide as an activating agent for CH_4 and natural gas storage at different pressures. Until now, limited research attention is given to the improvement of mechanical strength of activated carbon for adsorbed natural gas application. The role of PEEK in this study is to improve the dimensional stability (mechanical properties) and gas uptake of the adsorbent. In addition, the aim of this work is to investigate the adsorptive characteristics of analytical grade CH_4 and NG commercially available in the Malaysian market onto the synthesized activated carbons using the volumetric technique. In addition, it is to study the kinetics of process based on CH_4 and NG adsorption and desorption rate as fast kinetics are crucial toward achieving fast adsorption and desorption of CH_4 and NG (Mason *et al.*, 2014).

1.2 Problem Statement

Activated carbon has been found to be undoubtedly the most popular and widely used adsorbent in adsorbed natural gas (ANG) system throughout the world and superior compared to zeolites, silica gels, and metal organic framework. This is so because, the precursors are cheap, abundant and unsophisticated, and at the same time, the adsorption capacity is tremendous (Mohammed *et al.*, 2014a; Nasri *et al.*, 2014b). Of all the precursors applied for the synthesis of activated carbon, coconut shell is one of the most widely used due to its availability in most parts of the world and effectiveness in gas phase applications.

At present, compression and liquefaction are the most matured system of storage. The drawback is, compression requires high pressure (200–300 bar) while liquefaction is impractical. A relatively low pressure of 30-40 bar is achievable by ANG to store nearly Compressed Natural Gas (CNG). However, development of viable adsorbent with the ability for adsorption-desorption without obvious adsorbent structural deformation has becomes a challenge.

Most of the research conducted by previous researchers for the production of activated carbon focused on the pore structural modifications and surface chemistry for enhanced methane adsorption without considering the importance of its mechanical property. Limited research attention is given to the improvement of mechanical strength of activated carbon for this particular application. Adsorbent viability would be determined by its ability to adsorb-desorb gas for several cycles. Report has shown that adsorption-desorption of CH_4 can cause deformation of adsorbent leading in reducing its mechanical strength (Zhou *et al.*, 2017). The deformation strength of the adsorbent varies from the initial adsorption pressure and the number of adsorption-desorption cycles (Zhou *et al.*, 2015). The higher the initial pressure of adsorption and the number of adsorption-desorption cycles the more obvious the adsorbent structure deformation (Liu *et al.*, 2015).

To illustrate this issue of the mechanical strength, composite activated carbon from coconut shell and polyether ether ketone were developed. Polyether ether ketone is an aromatic, linear and semicrystalline thermoplastic with excellent mechanical properties. It was reported that polyether ether ketone porous carbon has good properties with high compressive strength and gas storage applications (McNicholas *et al.*, 2010). However, the high price of polyether ether ketone limited its commercial applications such as in gas energy storage. An alternative was initiated by developing adsorbent for adsorbed natural gas system from a readily available precursor in Malaysia (coconut shells) with polyether ether ketone to synergistically withstand high-pressure impact of several cycles of adsorption-desorption with an improved gas adsorption uptake.

To the best of my knowledge, no sufficient research on: Synthesis of composite sorbent from coconut shells and polyether ether ketone with KOH as an activating agent and optimization by Response Surface Methodology (RSM) followed by microwave activation to improve its mechanical properties and specific surface area. In addition, the potential effects of kinetics and adsorption isotherm models on the CH_4 and NG adsorption and desorption rates were evaluated as fast kinetics are factors for achieving fast adsorption and desorption rate.

1.3 Hypothesis

i. NG uptake is related to activated carbon preparation conditions.
ii. Lowering adsorbent temperature during gas (CH_4 and NG) adsorption process favors its adsorption uptake.
iii. Activated carbon stability at several adsorption and desorption cycles depends on the mechanical strength of its precursors (coconut shells and PEEK).

1.4 Research Objectives

The objectives of the research are:

i. To synthesize and characterize activated carbon (AC) from coconut shell and PEEK modified AC adsorbents.
ii. To investigate the adsorptivity of the characterized adsorbents toward CH_4 and NG gases.
iii. To study the isotherm and kinetics models of the adsorption.
iv. To examine the stability of the adsorbents

1.5 Scope of Study

The scope of this research work is limited to the following:

i. The precursors (coconut shell and PEEK) were carbonised followed by modification with KOH then activation using microwave equipment for enhanced methane and natural gas uptake. The characterization of the AC covers mechanical properties, functional groups determination, pH determination, and elemental composition determination, thermogravimetric analysis (TG, DTG, fixed carbon, ash and volatile matter), density, morphology and texture properties (scanning electron microscopy and specific surface area).
ii. Adsorptivity of the synthesized adsorbents towards methane and natural gases were investigated using the static volumetric technique, by pressure and temperature measurement across the adsorption and load cells. The methane and natural gas adsorption pressure range is 5 -35 bar, while the temperature is at an ambient and 5 ^{o}C. The methane and natural gas adsorption at temperature 5 ^{o}C achieved at pressure 5, 17 and 35 bar only.
iii. Kinetics of the adsorption was described using pseudo-first-order, pseudo-second order, Elovich and Weber-Morris models. The methane and natural gas adsorption equilibrium data were also correlated with Langmuir, Freundlich, Sips and Toth's isotherm models.
iv. Stability of the activated carbon was determined after seven successive of adsorption-desorption cycles. Normal adsorbed natural gas materials are tested for methane and natural gas storage at pressure range of 35 to 40 bars. But due to safety, the pressure used in this study is 35 bar only. Adsorbent desorption at this pressure was also evaluated.

1.6 Significances and Original Contributions of This Study

Coconut shell is an agricultural waste and abundant in Malaysia, has less economic value and their disposal is costly, while burning will equally cause damage to the atmosphere by the emission of greenhouse gases. Using the by-product for development of gas adsorbent can serve as a way of minimizing the negative environmental effect of this waste. In addition, it adds value to the agricultural waste, reduces the cost of its disposal, and provides a potentially cheap alternative to existing high cost of AC. An adsorbent with sufficient working capacity produced from coconut shell would go a long way economically and technically for competitive comparison with compressed natural gas conventional storage method. NG vehicle represents a cost-competitive, lower-emission alternative to the gasoline-fueled vehicle. The immediate challenges that confront the natural-gas vehicle are high-pressure system and bulky natural gas storage cylinders. Activated carbon is use as an adsorbent to store natural gas for vehicular application at lower pressure system and reduced bulky confined space.

1.7 Thesis Structure and Organization

This thesis comprises of five chapters. In each chapter, the relevant subjects discussed as follows:

Chapter 1 introduce the background of the study discussed in this chapter. The problems prompted this study was also highlighted. The objective, scope, and significance of this research were also stated.

Chapter 2 contain the critical review of the relevant literature to this research was made. Extensive discussion on Policies and regulations governing the greenhouse gas emission made in this chapter. The discussion on the importance of NG utilization as an alternative to conventional liquid fossil fuel also reviewed. The literature on activated carbons production methodology steps such as; carbonization, Modification, Activation with microwave, gas adsorption, and desorption was reviewed. The review on the use and importance of design expert software for optimization of activated carbon preparation variables is been highlighted.

Chapter 3 consists of a selection of equipment and materials, the methodology employed and steps followed in accomplishing the objective of the research work was stated. The steps include; carbonization, impregnation/modification of the produced bio-char, characterization of the raw precursor, biochar and the produced activated carbons, activation process with microwave and adsorption process. Adsorption isotherms and kinetics equations used in correlating with the experimental data. Also, the use of design expert software for optimization of parameters for preparation of activated carbons discussed.

In chapter 4, the results obtained for bio-char synthesis, activated carbon synthesis, characterization of the raw precursor, biochar and activated carbon were discussed. Adsorption of CH_4 and natural gas onto the synthesized activated carbon were also discussed.

Finally, in chapter 5, the findings of this research work were been presented. The recommendations were been made on how to improve for future work

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